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VOLUME 28 September 1951 NUMBER 9

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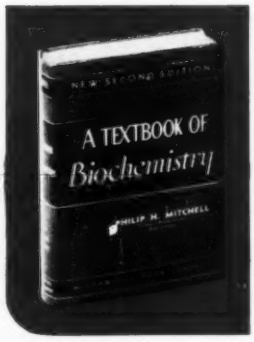

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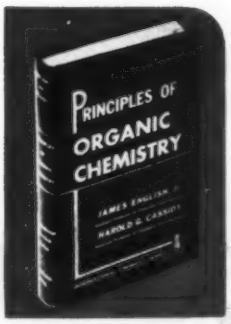

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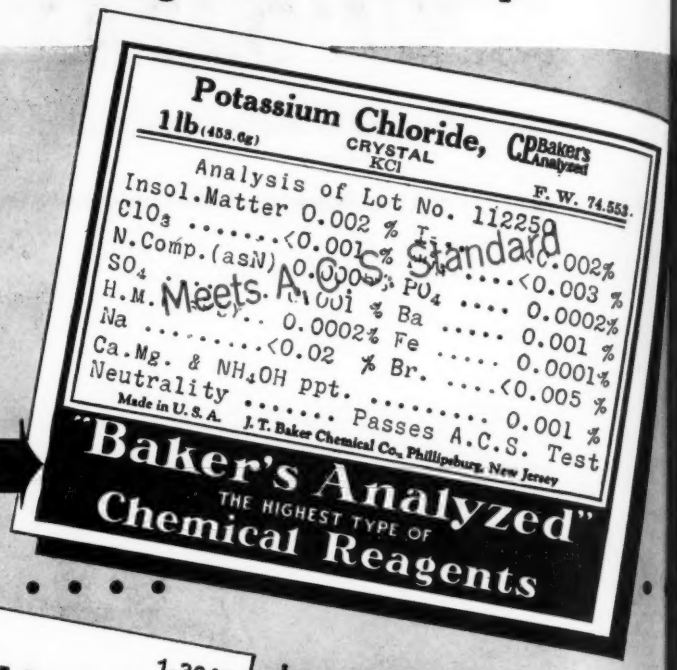

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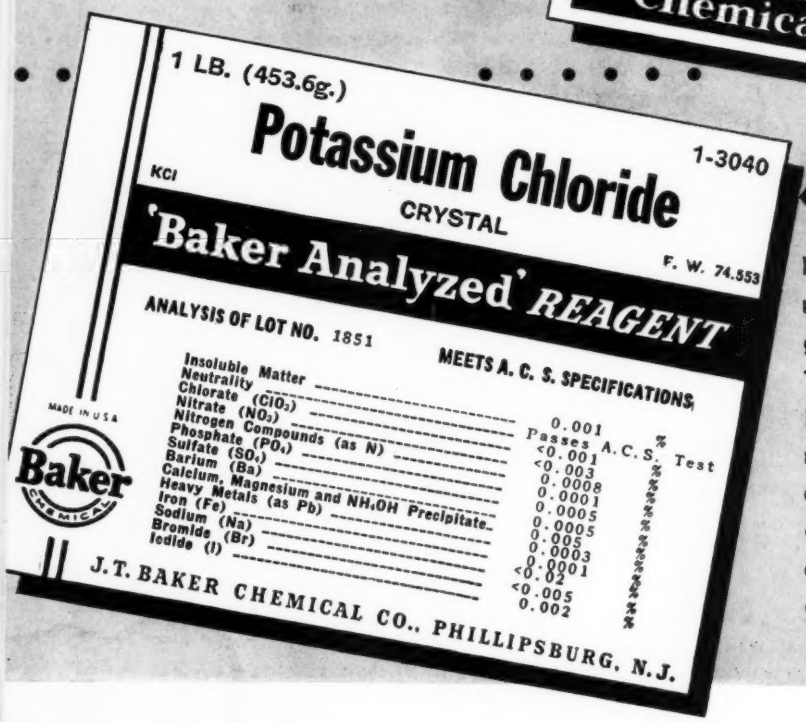

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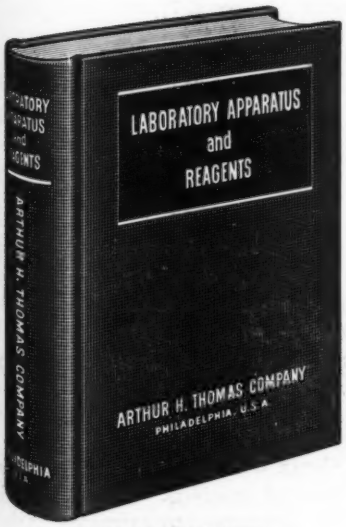

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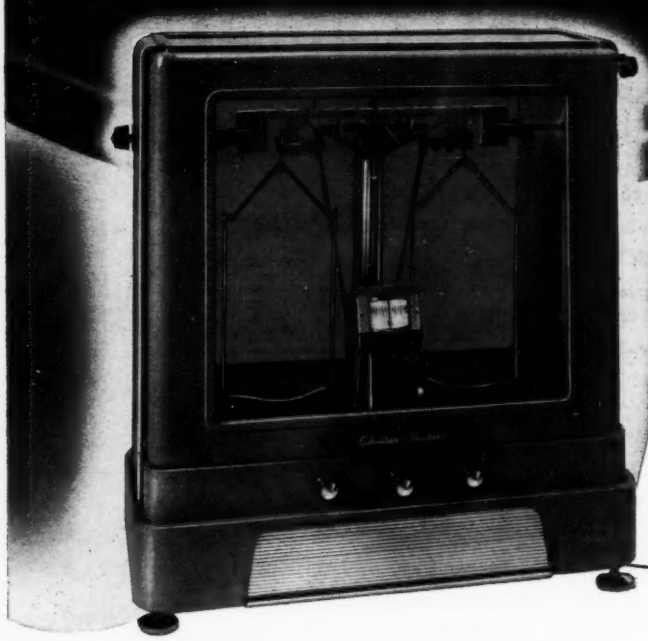

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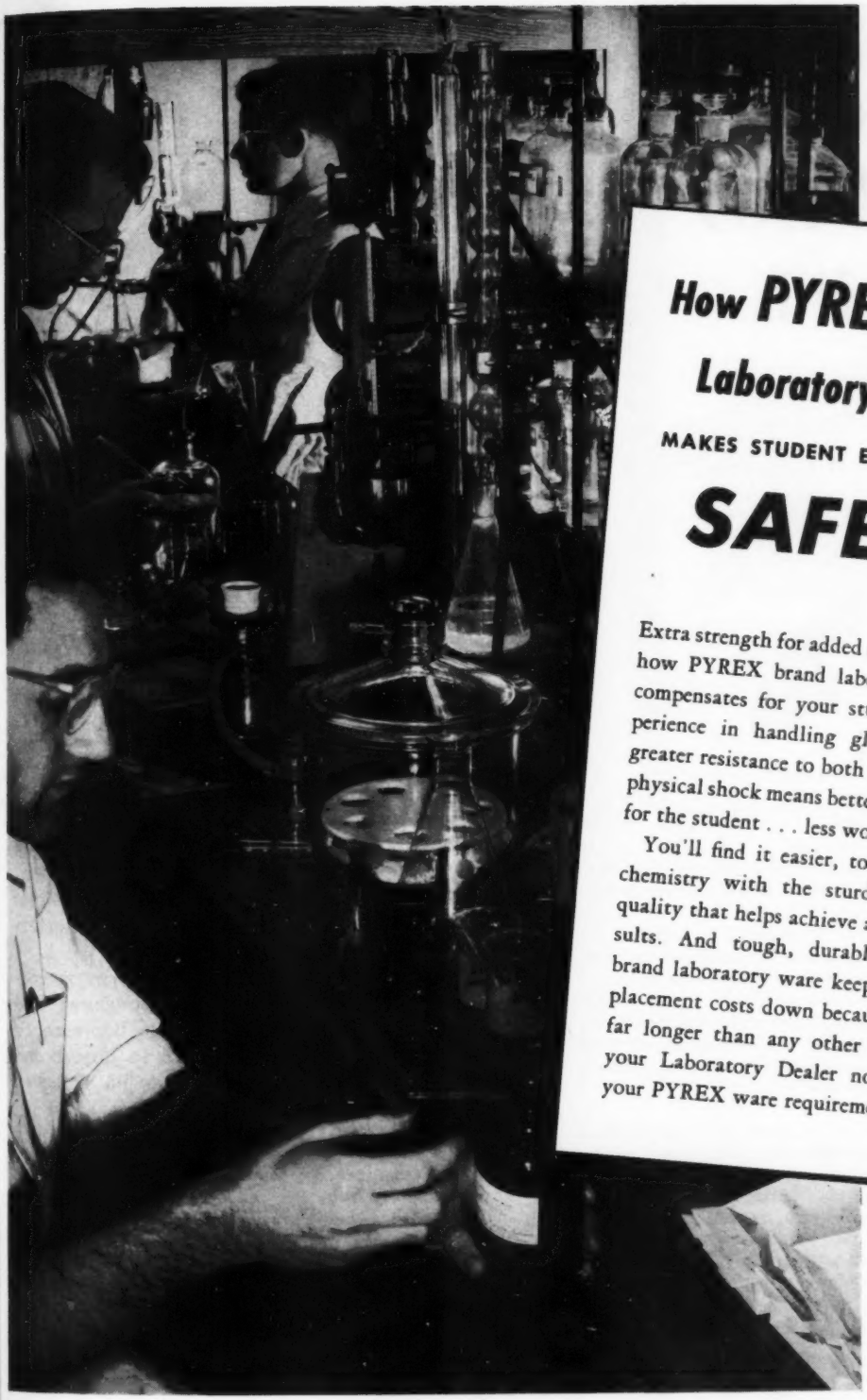

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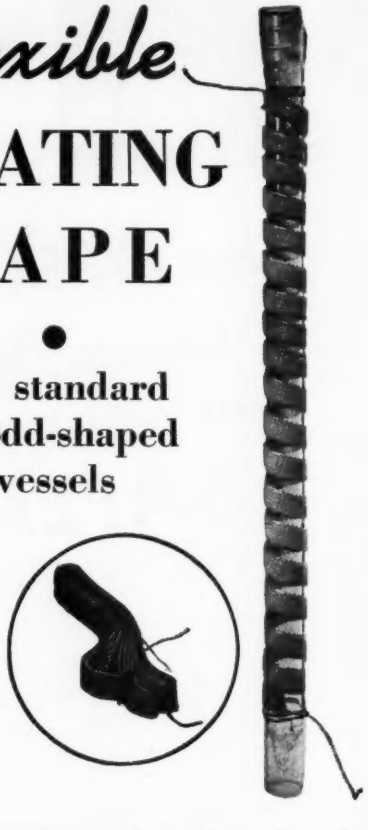

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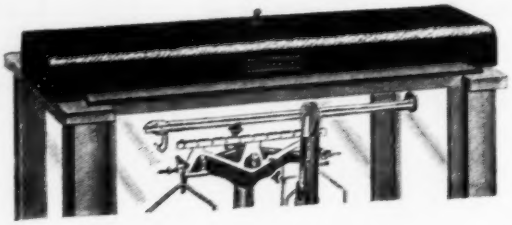

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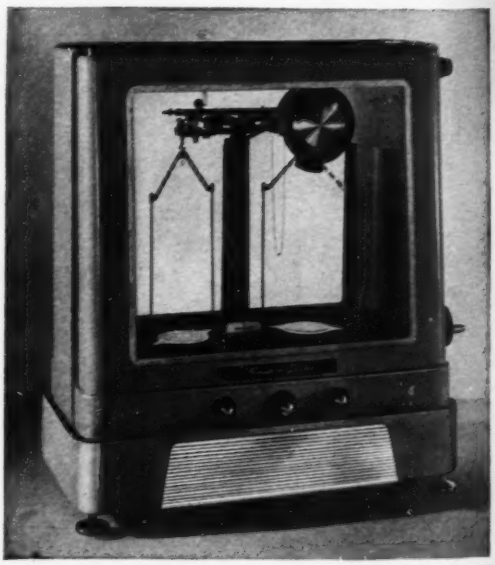

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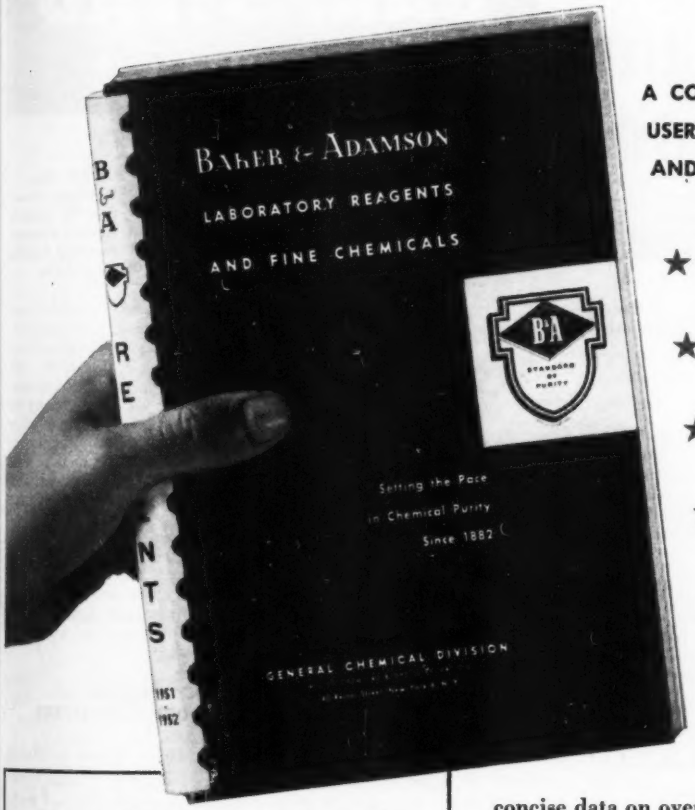

A COMPLETE BUYERS' GUIDE FOR USERS OF LABORATORY REAGENTS AND FINE CHEMICALS

- ★ For Manufacturing Control Laboratories
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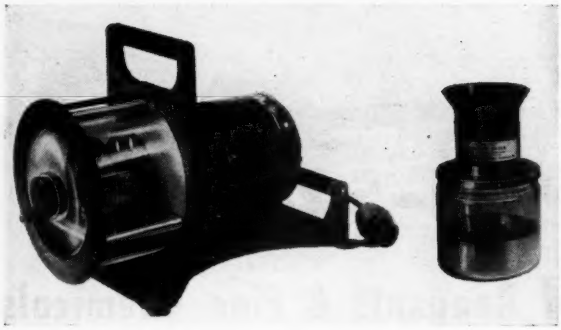

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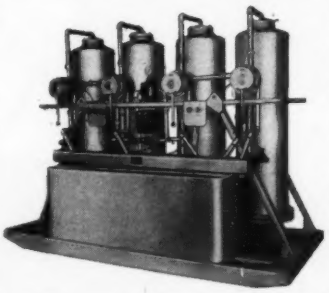

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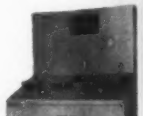

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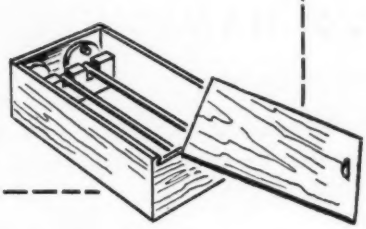

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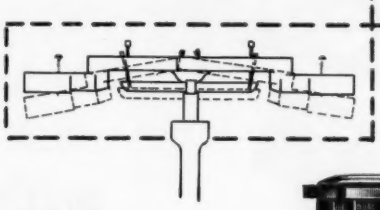

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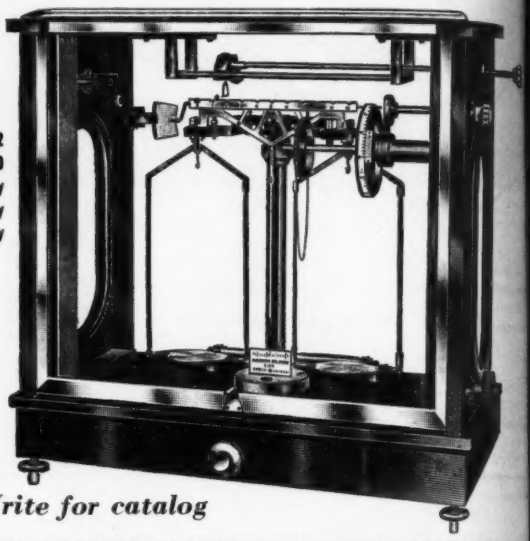

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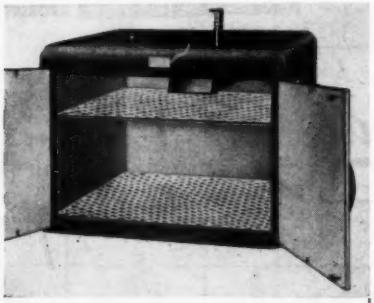

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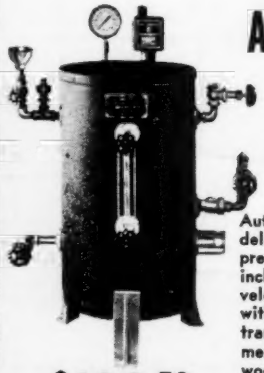

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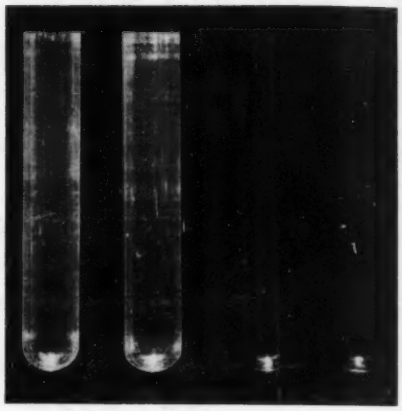

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Dural H is designed for washing laboratory glassware by hand. And Dural H is safe. It is safe on glass because it is inhibited to prevent early etching, weight loss and premature breakage. It is safe and easy on the hands because it is unusually mild.

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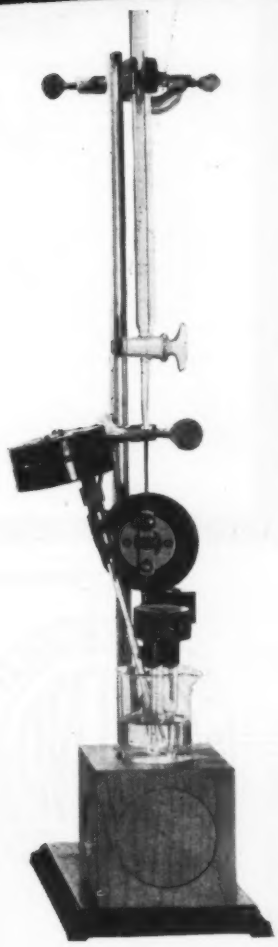

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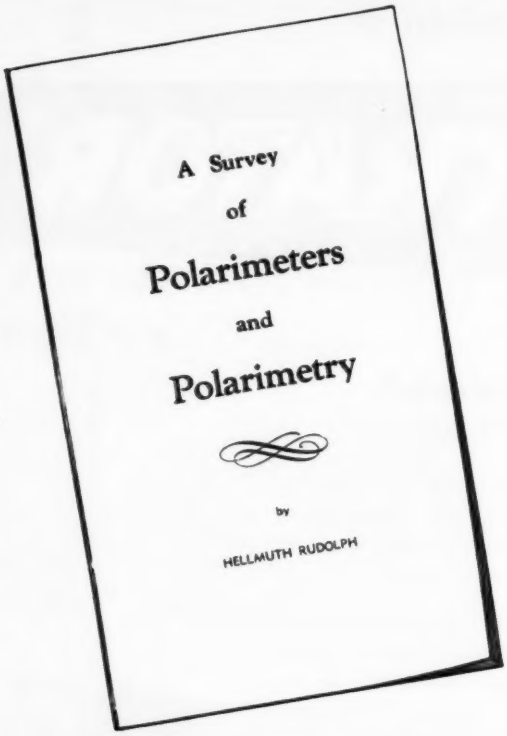

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accuracy . . . speed . . . simplicity

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Spectrophotometric measurements—based on analysis of liquid, solid or gaseous samples by light absorption—have become one of the most important methods of today's process control operations. Complicated analyses requiring hours to perform by standard chemical procedures are now completed in a matter of minutes using modern spectrophotometric methods.

Up to the present, *quality* instruments have been beyond the reach of many laboratories. Now, however, the development of the new Beckman Model "B" Spectrophotometer makes available for the first time a *precision* instrument—convenient and simple to operate—*yet versatile, accurate and low-priced.*

The Model "B" combines many of the advancements pioneered in the famous Beckman Quartz and Infrared Spectrophotometers . . . high resolution . . . wavelength and photometric accuracy . . . freedom from stray light . . . wide spectral range . . . quality construction.

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The Model "B" is by all standards the outstanding instrument in its field. Write for full details. *Beckman Instruments, National Technical Laboratories, South Pasadena, 36, Calif.*

Why Spectrophotometric Analytical Methods Demand Classroom Attention

Because spectrophotometric methods are frequently faster, simpler and more accurate, they are being increasingly adopted by all types of industrial and research laboratories. **In a leading chemical publication over 30% of all 1948 editorial matter dealt with modern spectrophotometric methods . . .** a significant indication of the vital importance of these methods in today's chemical field.

Leading colleges and universities, recognizing the importance of stressing new instrumental analytical methods, are including laboratory work with spectrophotometers in basic quantitative analysis courses. These methods also permit better and faster study of many physical chemistry principles and problems, such as photochemistry, equilibrium constants and reaction rates, thus allowing increased time for these and other subjects.

In instruments for student use, quality is of paramount importance to insure a true appreciation for the usefulness and reliability of the methods studied. The Beckman "Model B" Spectrophotometer is ideal for student work because it is a **quality** instrument throughout—accurate, versatile, rugged—yet priced within even the most modest budget. Moreover, its simplicity of operation, its direct-reading scales, its full range from ultraviolet to near infrared—all make this instrument unsurpassed for educational work. And the wide range of accessories further facilitate adequate student instruction in the many phases of spectrophotometric applications.

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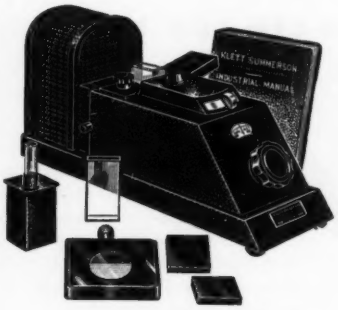

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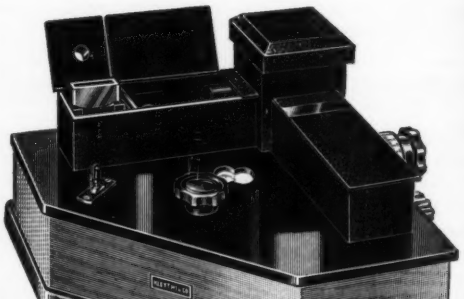

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MODEL III POLAROGRAPH

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MANUAL

E. H. Sargent & Co., the manufacturer of Polarographs, offers three different Polarographs—the Model XXI Visible Chart Recording; the Model XII Photographic Recording, and the Model III Manual.

The Model III Polarograph may be used in any phase of polarography, but it is particularly recommended for applications to which it is peculiarly suited and in which recording facilities are unnecessary.

The Model III is recommended:

(1) In those routine analyses where only one substance is determined and where the step shape approximates a pure form, characteristic of reversible reactions involving simple ions in fairly substantial concentrations. In such cases, step height may be measured as the difference between two points, one on the residual current plateau and one on the diffusion current plateau. For measurements of this type, a simple manual instrument such as the Model III is preferable to a recording Polarograph because of the speed and ease with which it may be operated even by relatively non-technical personnel.

(2) In those laboratories where a recording instrument is now in use for several determinations, a Model III may be used for each determination, thus releasing the recording Polarograph for research purposes and for establishing the conditions of the routine analyses.

(3) In the performance of amperometric titrations.

(4) For instructional purposes in educational institutions where the 315 mm. long, readily visible scale and the simplicity of design make the Model III Polarograph ideal for demonstrations and illustration of the basic circuit employed in Polarographs.

Essentially the Model III provides facilities for the incremental application of voltage across the dropping mercury electrode cell and for indicating the resultant current passing through the cell. The cell current is measured by a highly sensitive galvanometer from which a line light image is transmitted, through a mirror system, to a translucent scale located above the instrument panel. The scale is double graduated from −150 to +150 mm. with zero center in red at top and from zero to +300 mm. in black at the bottom. The scale is 31.5 cm. long, with circular curvature, to eliminate tangent error. Each graduation is extended 15 mm. at left for zero adjustment. A continuous selection of bridge voltage from 0 to 3 volts is provided, and the selection of output voltage is by rotation of a single dial, this dial reading direct in millivolts per span volt. Current is calculated simply by multiplying scale reading by multiplier reading by sensitivity coefficient of galvanometer. To adjust galvanometer sensitivity a ten position Ayrton galvanometer shunt is provided. Galvanometer sensitivity is approximately 0.005 microamperes/mm. and galvanometer shunt ratios are from 1:1 to 1000:1. A three position toggle switch permits that the sense of the galvanometer scale remain unchanged regardless of cell current direction or polarity of the dropping electrode.

A ten turn helical potentiometer serves both for upscale and downscale compensation and for adjustment of the galvanometer zero position.

Another potentiometer incorporating a power switch serves to select the desired voltage across the bridge. A panel mounted voltmeter accurate to 1.0% of full scale indicates the voltage established.

The instrument is mounted on a cast aluminum base and enclosed in a sheet metal housing provided with a hinged top and finished in black enamel. All controls are mounted on a polished aluminum panel.

S-29290 POLAROGRAPH—Manual, Indicating, Model III, Sargent. Complete with three No. S-30858 dry cells, one No. S-29304-A galvanometer lamp, one set No. P.N. 1995 cell lead wires, one No. P.N. 2067 calibrating resistor plug, 250,000 ohm, and cord and plug for connection to standard outlets. For operation from 115 volt 50 or 60 cycle circuits.....**$390.00**

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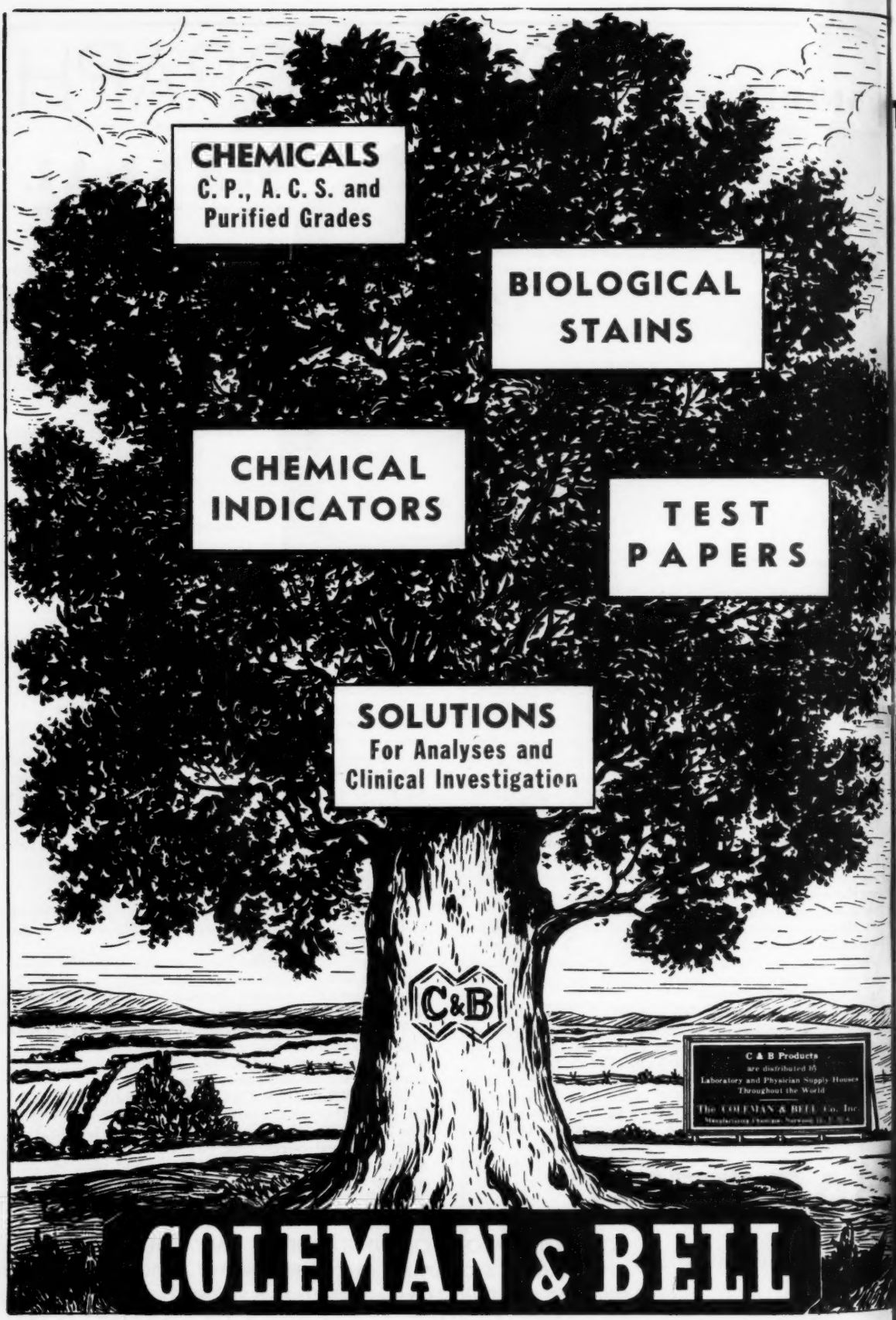
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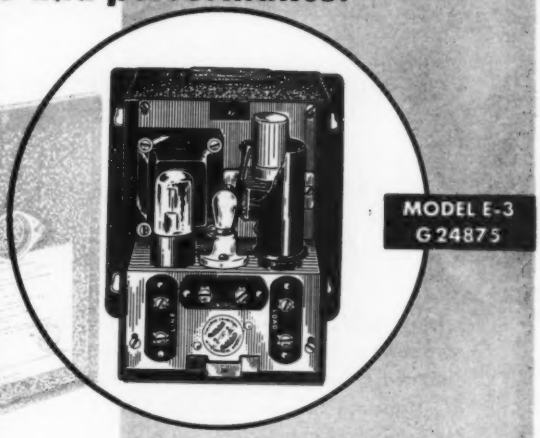
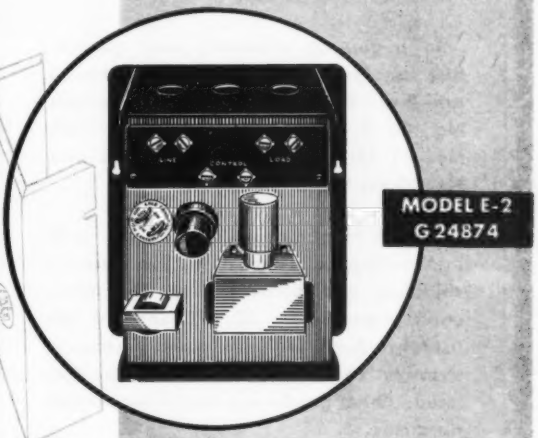
**See article by David L. Shapiro in Radio Electronic Engineering, June 1948.*





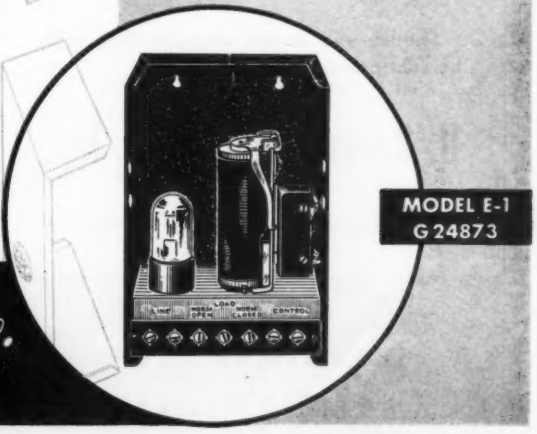


TABLE OF RELAY CHARACTERISTICS			
Model No.	**E-1**	**E-2**	**E-3**
Load Current	10 amp. 115 V. A.C.-D.C.	30 amp. 115 V. A.C.	30 amp. 115 V. A.C.
Relay Type	Microswitch	Mercury to Mercury	Mercury to Mercury
Control Voltage	35 V. A.C.	6.3 V. A.C.	16 V. D.C.
Control Current	15 Microamp	6 Microamp	3 Microamp
Max. Control Lead Length	25 feet (cable of 30 $\mu\mu f$/ft.)	40 feet (cable of 30 $\mu\mu f$/ft.)	No Limit
Tube Complement	One 35L6	One 2D21	One 6SN7 One 6L6
Dimensions	6½"H x 5⅛"W x 4"D	7⅛"H x 7"W x 3⅜"D	8"H x 7¼"W x 5¾"D
Weight	4½ lbs.	6 lbs.	7½ lbs.
Mounting Provision	Key holes in back of case	Key holes in flanges	Key holes in flanges

Features: E-2 and E-3 have jumpers for selecting either normally open or normally closed operation. E-1 has both types of contacts on the microswitch.

Price List

G24873	E. G. Co.	Electronic Relay Model E-1	ea. 35.00
G24874	E. G. Co.	Electronic Relay Model E-2	ea. 45.00
G24875	E. G. Co.	Electronic Relay Model E-3	ea. 62.50

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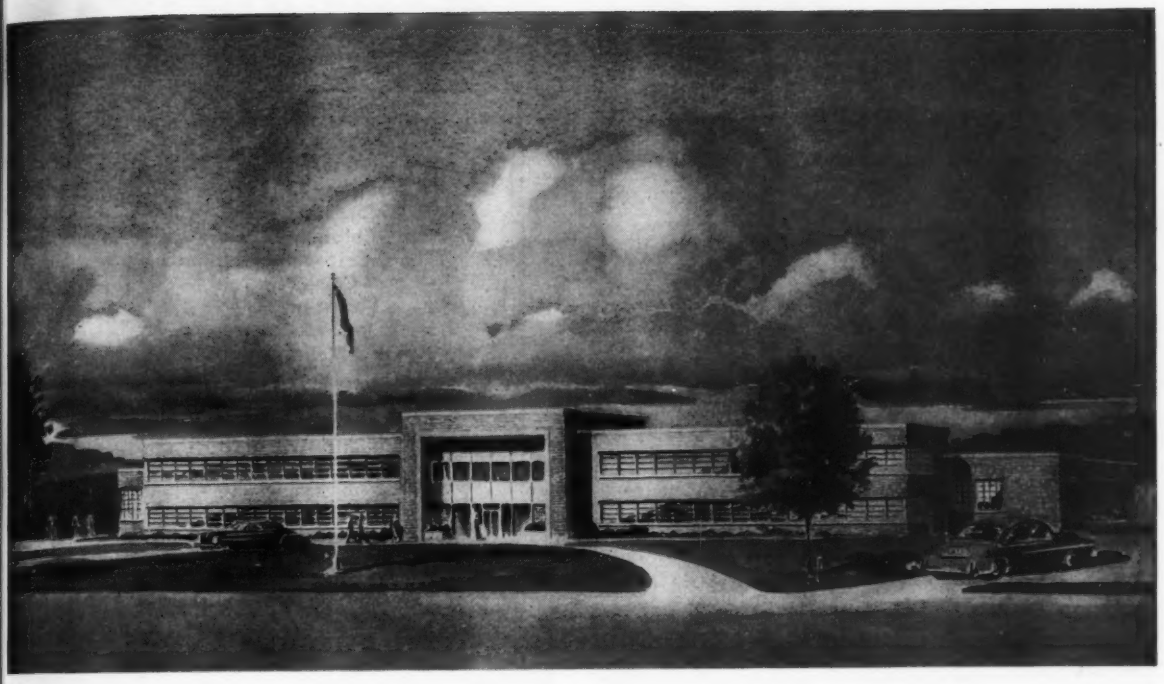

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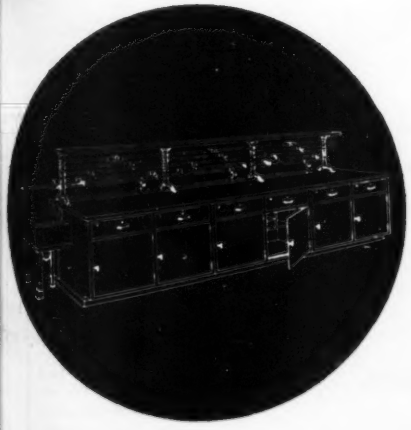

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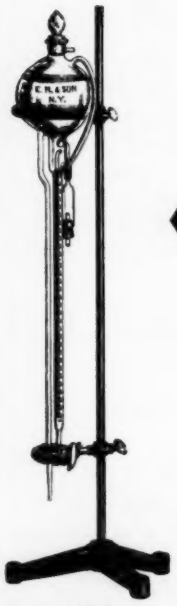

8-526

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Reservoir cap., ml.	500	1000	1000	1000	2000	2000
Each	$22.50	23.75	23.00	25.00	27.00	28.50

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Pipette capacity, ml.	5	10	25	50
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Each	$20.50	21.50	23.00	24.00

Write for Bulletin A-B

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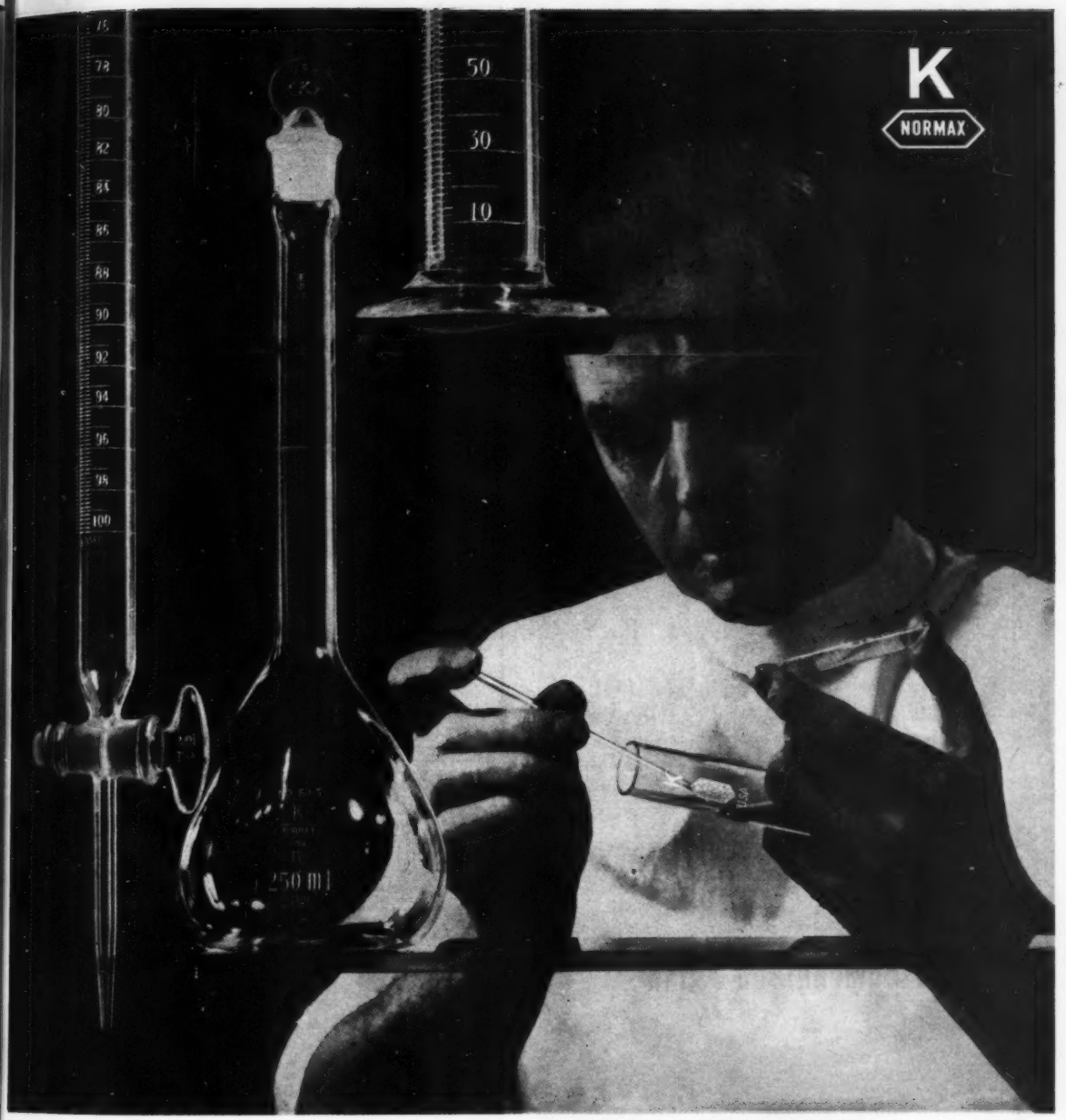

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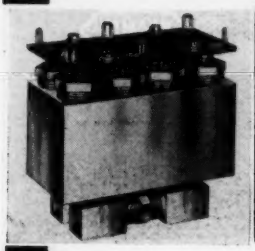

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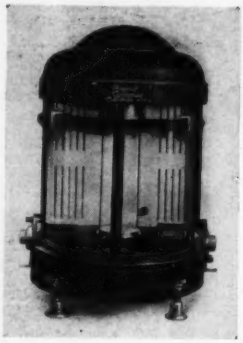

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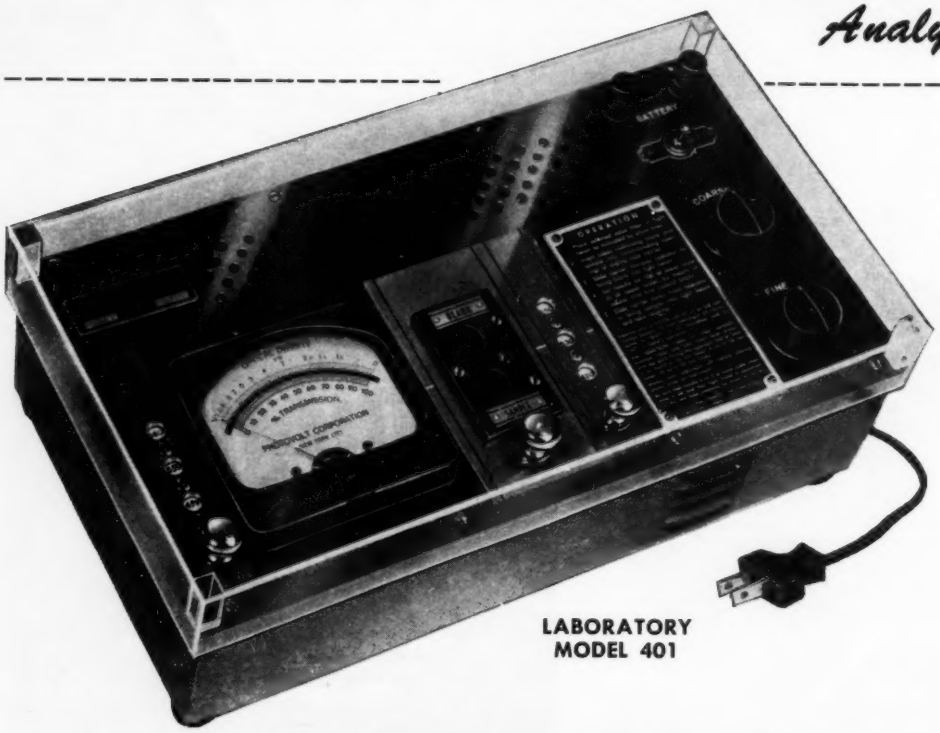

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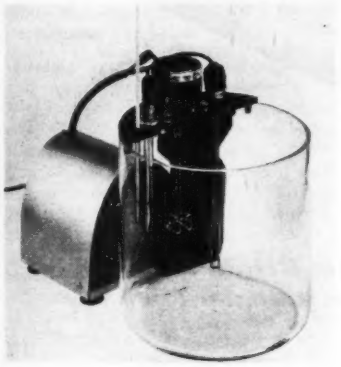

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The range of the bath as regularly provided is from room temperature to 55°C. but can be extended either by the use of auxiliary heaters or by means of a cooling tube.

Current Research

Scientific research done by the nation's engineering schools is the subject of a new book called "Review of Current Research," published by the Engineering College Research Council of the American Society for Engineering Education. The review outlines the policies and activities of engineering research in the 91 colleges and universities holding membership in the F.C.R.C., and is a complete guide to the current research contributions of these engineering schools.

In addition to research project titles, the book shows for each school the names of responsible research administrative officers, a brief digest of policies which govern research projects and contracts, the number of personnel engaged in research activities, the annual expenditures, and special conferences and short courses of interest to research workers. The listings are complete as of March 1, 1951.

The publication may be obtained from the Engineering College Research Council, Room 7-204, 77 Massachusetts Avenue, Cambridge 39, Massachusetts.

Gas Price List

A complete up-to-date descriptive listing of laboratory gas prices is now obtainable from the Ohio Chemical & Surgical Equipment Co., 1400 East Washington Avenue, Madison, Wisconsin.

In addition to prices, the informative reference folder includes a tabulation of the basic properties of laboratory gases, as well as a summary of an analysis of principal impurities.

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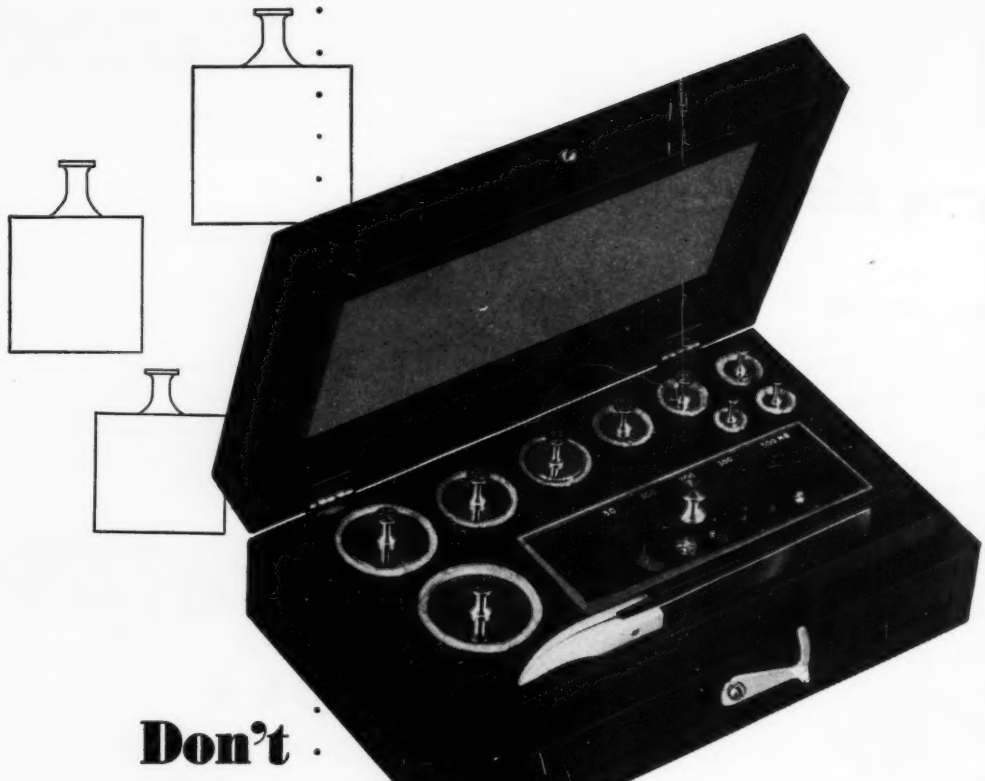

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Literature Available

Colleges offering courses in instrumentation for quantitative analysis, may be interested in time-saving analytical methods being used in industrial applications. Many new procedures are replacing older, classical methods.

For laboratory workers who are interested, Central Scientific Company has compiled a bibliography of analytical methods in which filter photometers and spectrophotometers are used. Called List "Bimet," the bibliography is a compendium of references to the more important articles appearing in current periodicals. Also available is List "Titration" containing suggestions for using a line-operated pH meter in various titration applications. Both lists may be had on request to Central Scientific Company, 1700 Irving Park Road, Chicago 13, Illinois.

A new Combustion Tube Furnace bulletin, LAB-1236-A, which will replace LAB 1236, is now available from Hevi Duty Electric Company, Milwaukee 1, Wisconsin. This illustrated bulletin describes the uses, construction, and specifications of Hevi Duty Combustion Tube Furnaces.

Coleman Autotrator, Model 19 for Automatic Titration

According to the manufacturer, this new Coleman instrument provides precise, high-speed, fully automatic titration at minimum cost, and with the least possible uses of space or time. The Autotrator performs automatically all the steps necessary to a perfect titrating technique. Titrant is delivered rapidly until the end point approaches, then is restricted, the final portions being added in progressively smaller increments until the true end point is reached. Operation is controlled by the Coleman Model 18 pH Electrometer which measures and continuously indicates the progress of the titration.

The Autotrator is designed to meet actual laboratory needs. It employs standard laboratory accessories and occupies a minimum of laboratory space.

Complete details are in Coleman Bulletin B-223, which may be had by writing Coleman Instruments, Inc., 318 Madison Street, Maywood, Illinois.

New Mechanically Convected Oven

The Electric Hotpack Co., Inc., has just announced a new table model mechanically convected oven with automatic temperature control which uses a minimum of table space. The interior dimensions of the chamber are 12 in. wide × 14 in. deep × 14 in. high, but the exterior dimensions are only 23 in. wide × 20 in. deep × 40 in. high. Thus the new Hotpack mechanically convected oven takes up 50% less table space than the ordinary comparable table model. The floor of the working chamber is just 9 in. above the table making it easy to load and unload, and all controls are located above and out of the way of the working chamber, yet within easy reach for adjustment.

The unit is constructed either of heavy gage steel exterior, finished in baked-on laboratory gray enamel, with a polished stainless steel interior, or entirely of noncorrosive 18-8 polished stainless steel. It has a blanket of 3 in. glass wool insulation surrounding the chamber on all sides and the heating elements are guaranteed for the life of the oven.

The range of the new oven is from 35° to 180°C. with a temperature differential of plus or minus $\frac{1}{2}$°C. It is equipped with an adjustable hydraulic control thermostat and the Hotpack Limitstat, an over-temperature thermal switch, which prevents the chamber temperature from exceeding the control setting.

More details are available from the manufacturer, The Electric Hotpack Co., Inc., 5097 Cottman Avenue, Philadelphia 35, Pa.

Lab-Lift

Easy positioning of heavy or hot laboratory equipment, such as reaction flasks, cold and hot baths, etc., at various levels above the workbench is afforded by use of a "Lab-Lift" device manufactured by the Fisher Scientific Co., 717 Forbes Street, Pittsburgh 19, Pennsylvania. It has a platform which can be adjusted to any point from 11 to 18.5 in. above the workbench by turning a screw-support to which the platform is attached. Both the movable platform and the base have upright cadmium-plated rods for holding clamps, thermometers, stirrers, etc. The device is claimed to be more stable and easier to operate than previous models or makeshift arrangements.

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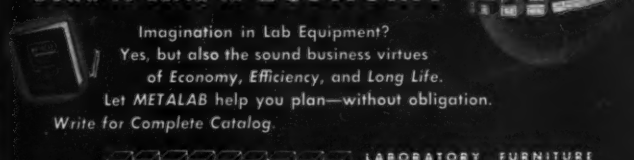

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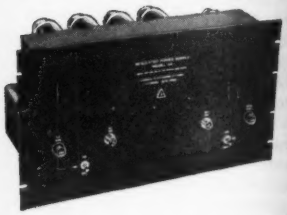

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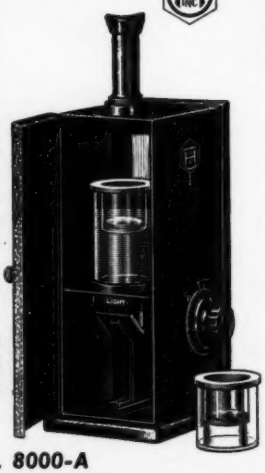

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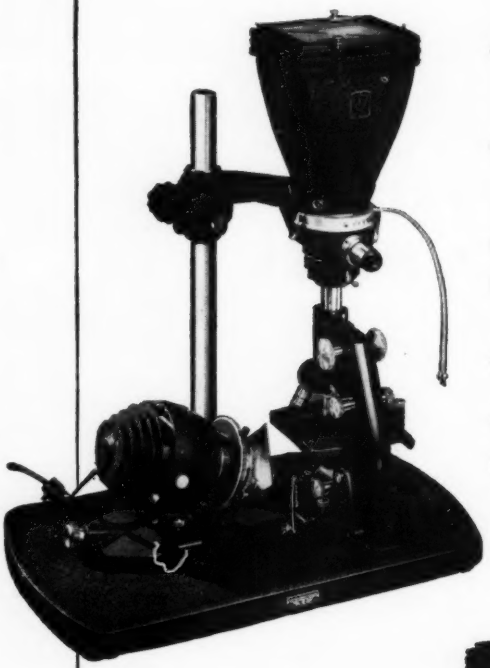

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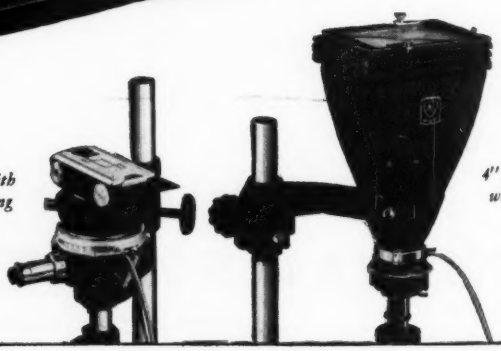

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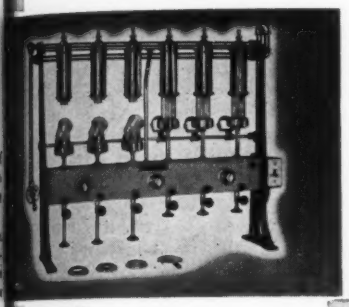

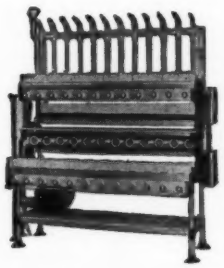

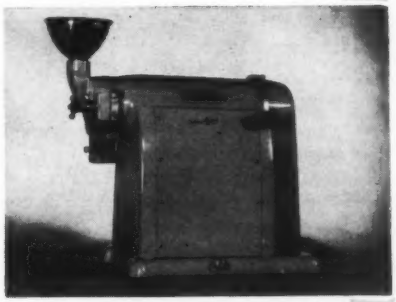

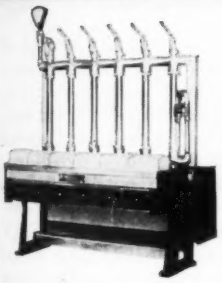

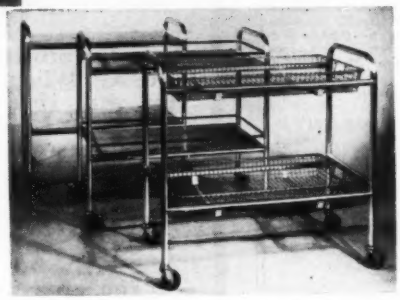

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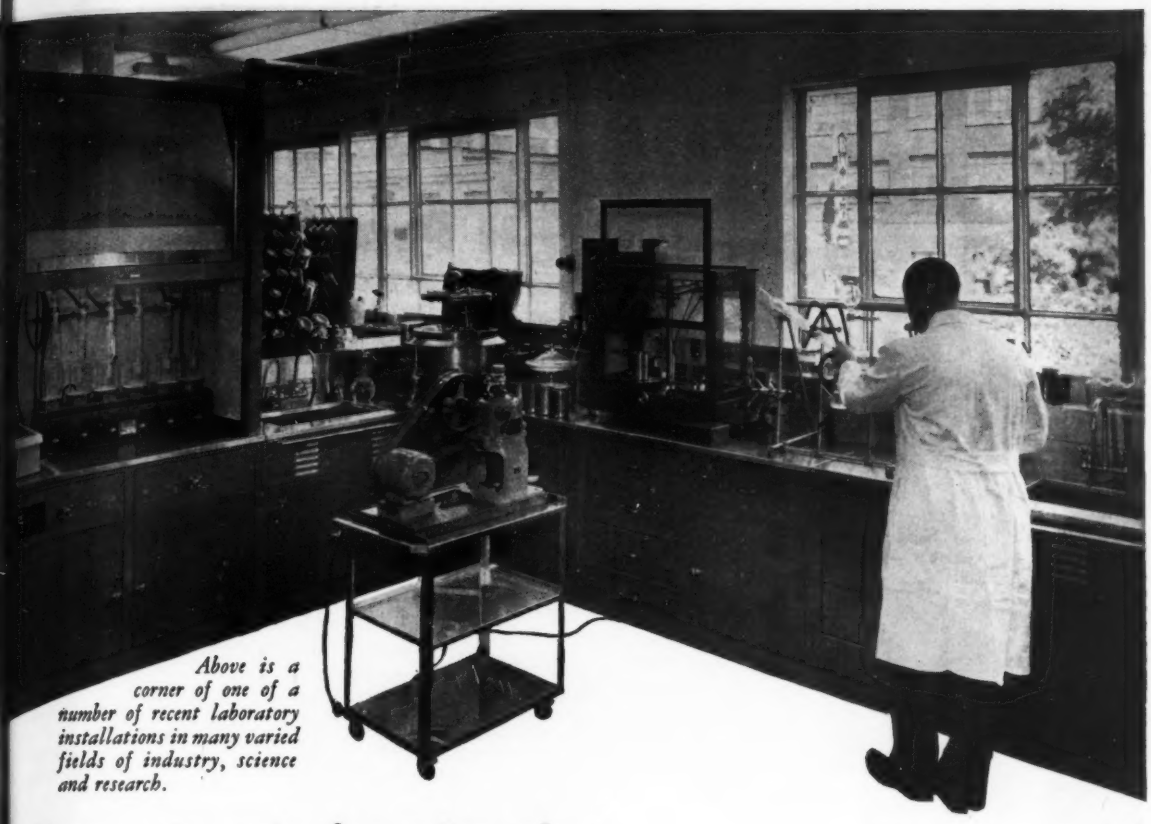

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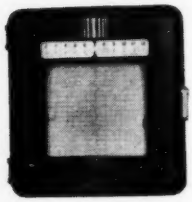

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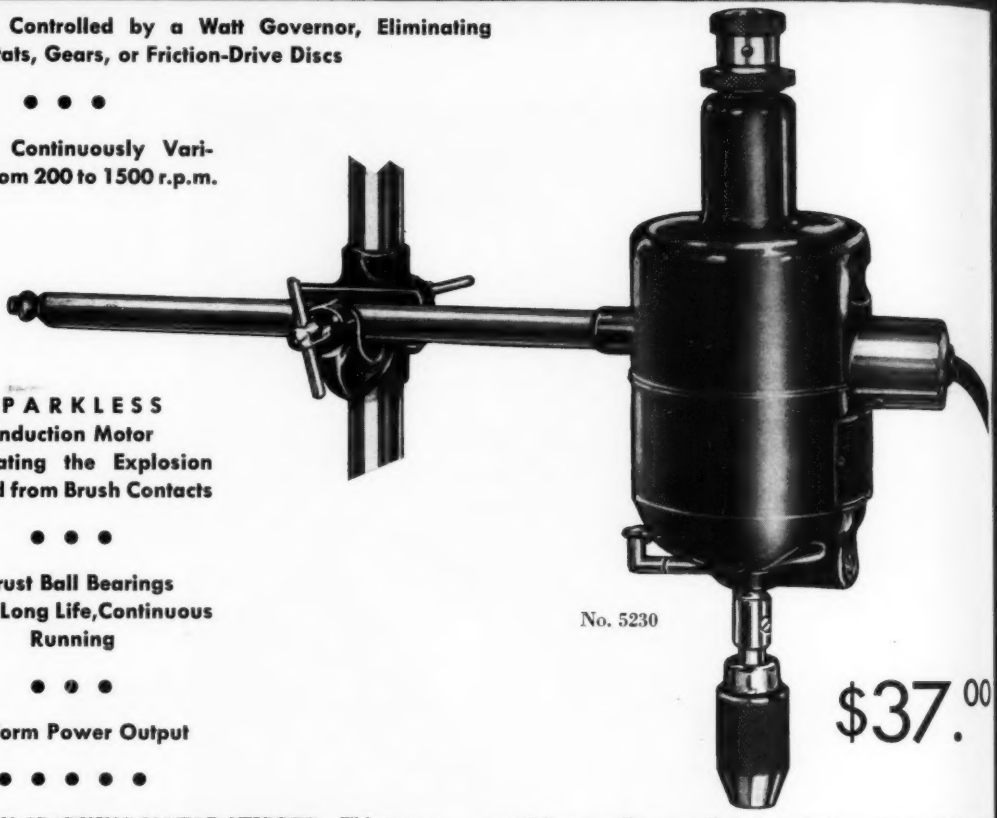

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Editor's Outlook

It is not often that I use this page to discuss family matters. However, in these days in which awards are given for all sorts of meritorious service there is one group of people who deserve praise and appreciation which they seldom get. I want to drop a few words of appreciation to them.

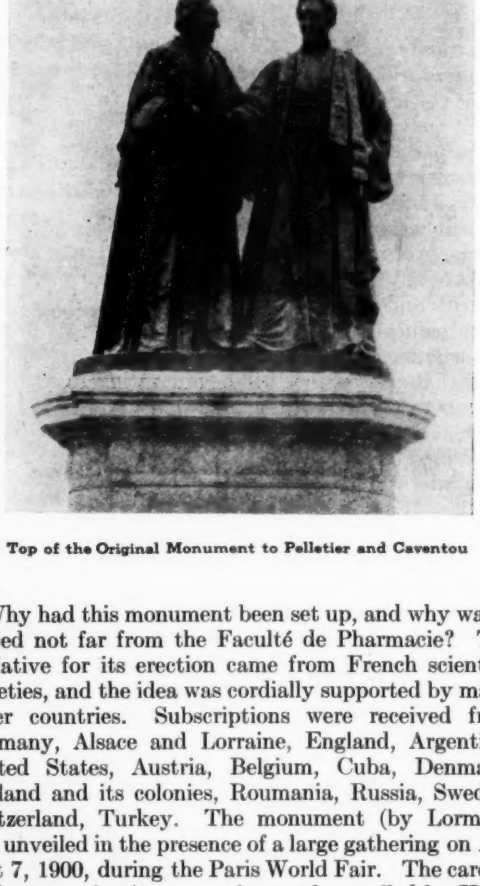
A publication such as This Journal, with a part-time, non-professional editor, cannot get along without a lot of help. I am sometimes amazed, always gratified, to discover how freely such help is given. This Journal does not have as complicated a "referee system" as do the professional research journals but it depends very definitely upon the advice and help of many of its friends in the choice and selection of its editorial material. Manuscripts of any size or potential importance are generally read by two or more competent and impartial reviewers before acceptance or rejection. The field of chemical education is so broad and diffuse that it is impossible for an article to be important or useful to all the readers—perhaps even to a majority. But we do expect that every article will be interesting and valuable to *some*. The editor cannot always trust his own judgment but must try to get the representative views of some of his friends.

Reading manuscripts for this purpose is a chore, although some of our reviewers are kind enough to say that they often find it interesting. But it takes time and must interfere with other more important duties. For the first few times there is perhaps a subtle feeling of flattery that one's opinion is respected enough to ask, but later one must certainly begin to wonder why he is continually being made "the goat." The fact that we seldom hear complaints must be evidence that a surprising number of people are seriously interested in the success of publications like ours and that they feel a certain professional responsibility for that success. In any event, such cooperation and community of interest is certainly very gratifying; indeed, we couldn't very well get along without it. If some of our collaborators should read this—as I hope they will—I want them to know that we are profoundly grateful.

JOSEPH PELLETIER AND JOSEPH CAVENTOU

MARCEL DELÉPINE
Paris, France
(Translated by Ralph E. Oesper)

Up to a few years ago in Paris, in the square where the Boulevard Saint-Michel and the Rues Auguste-Comte, Denfert-Rochereau, and Abbé de l'Epée come together, there stood a striking monument. Its two bronze statues depicted Pelletier and Caventou in academic dress. Only the stone pedestal of this memorial remains; during the German occupation the figures were removed and the metal was melted down, presumably for the manufacture of copper shell bands.



Top of the Original Monument to Pelletier and Caventou

Why had this monument been set up, and why was it placed not far from the Faculté de Pharmacie? The initiative for its erection came from French scientific societies, and the idea was cordially supported by many other countries. Subscriptions were received from Germany, Alsace and Lorraine, England, Argentina, United States, Austria, Belgium, Cuba, Denmark, Holland and its colonies, Roumania, Russia, Sweden, Switzerland, Turkey. The monument (by Lormier) was unveiled in the presence of a large gathering on August 7, 1900, during the Paris World Fair. The careers of the two scientists were eloquently recalled by Henri Moissan, chairman of the subscription committee, and Leon Guignard, director of the École Supérieure de Pharmacie (since 1924 the Faculté de Pharmacie).

Since the war a new monument in honor of Pelletier and Caventou has been made. It was unveiled on March 2, 1951, and stands on the site of the former one. The artist wished to represent "la santé recouvrée" as a token of gratitude to the discoverers of quinine, whose medallions are placed on the sides of the pedestal.

This paper will give a short account of the exemplary lives of these two scientists and their accomplishments. Even though the discovery of quinine was the capstone of their careers, it should be noted that it was only one step in the course of their many successful and notable researches.

Joseph Pelletier was the grandson of Bertrand Pelletier, pharmacist at Bayonne, pupil and friend of the illustrious G. F. Rouelle (1703–70), who was also the teacher of Lavoisier. The father (1761–97), who likewise was named Bertrand, was also a pharmacist. His professional career was spent in Paris, where guided in the beginning by D'Arcet (Rouelle's son-in-law) he published some nice researches on tin, phosphorus and its derivatives, extraction of salt, etc. He was a member of the old Académie des Sciences, professor at the École Polytechnique, and member of various scientific or military commissions during the revolutionary period. Therefore, it is clear that Joseph Pelletier, who was born on April 22, 1788, in Paris, was raised in an atmosphere highly favorable to the development of any scientific aspirations, which a scion of such ancestors would presumably inherit.

After Rouelle died his widow assisted by her son and brother carried on the Rouelle pharmacy on the Rue Jacob until 1779. In 1794 she sold the business to Bertrand Pelletier, who had been her *proviseur* (manager) during the year before.

Joseph Pelletier began his pharmaceutical training in 1806 and quickly showed his excellence as a student. In 1807 he received the highest award in chemistry from the hands of Fourcroy, and the next year obtained similar prizes in botany and natural history. Such academic honors are well calculated to engender a student's self confidence, and often are the prelude to a brilliant career.

Pelletier's exceptional intelligence enabled him to earn quickly the degrees necessary to a teaching post in the École de Pharmacie. Pharmacien in 1809, Docteur ès Sciences in 1812, he was called in 1814, when only 26, as *adjoint* professor to Robiquet, who held the chair of natural history. Pelletier, as a former pupil of Haüy, was given charge particularly of mineralogy, but when Robiquet in 1824 was appointed administrator-treasurer of the school, Pelletier succeeded to his academic

position and lectured on the materia medica of all three natural kingdoms. He conducted his course for 15 years (1825–40) with much success, since the students were captivated by his animated and eloquent delivery. In addition, he took an active part in administrative affairs, and he must be counted, along with his friend Caventou, among those who little by little brought the reputation of the École up to the level of the other faculties.

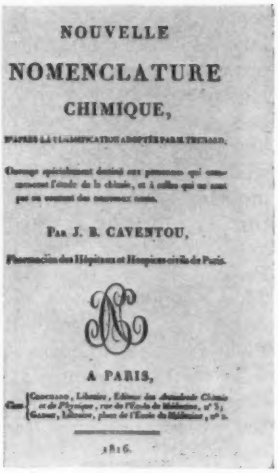
Pelletier's rapid advancement to the foremost posts in the teaching of pharmacy was doubtless a result of the prestige attached to his discoveries, whose merit was unhesitatingly acknowledged. He was elected to the Académie de Médecine in 1820, and became free associate of the Académie des Sciences in 1840. Neither his official post as professor nor his honors prevented him from actively practicing his profession; his apothecary was in the Rue Jacob.

Pelletier died on July 19, 1842, the victim of a cruel malady. His Christian resignation and philosophical serenity concealed his sufferings, because he did not wish his dear ones to suspect his excrutiating pain. The celebrated chemist, J. B. Dumas, who was then vice-president of the Académie des Sciences, pronounced the funeral oration in terms which, issuing from his eloquent lips, glorified the life and work of the illustrious deceased, who had been his friend and on occasions his collaborator (*1*).

Joseph Bienaimé Caventou came from a Poitou family; the grandfather was a master coppersmith at Poitiers. His father did not continue in this grade but, doubtless swept away by the patriotic impulse of the moment, he entered the armed services to become a pharmacist. Even at that time, the military pharmacy could point with pride to the reputations of some of its members, *e. g.*, Brongniart, Dizé, Cadet de Gassicourt, Parmentier. Caventou's father followed honorably in their footsteps, and his scientific accomplishments won him the title of correspondent of the Académie des Sciences. After serving in the armies of the Republic, he was assigned to the military hospital at Saint Omer, the capital of Pas de Calais. He married there and made this town his permanent residence. His son Joseph Bienaimé was born there on June 30, 1795.

After finishing the lower schools, Joseph began to study pharmacy with his father, but the latter decided that the young man ought to complete his training in Paris. In his memorial lecture of Caventou, Bergeron (*2*) tells of the journey of the young student. Leaving Saint Omer with very little in his purse, he decided to walk to the capital, a distance of more than 100 miles. Fortunately, before he had gone far, a fishcarrier, who was taking his wares from Dunkerque to Paris, offered him a place in the back of his cart, and so he arrived at his destination much sooner than he had dared hope.

He lodged with a pharmacist who was a friend of his father, and attended classes at the Faculté des Sciences and the École de Pharmacie. In 1815, he competed for the interneship in pharmacy and was awarded first place but, filled with patriotic fervor at Napoleon's return from Elba, the young man (he was only 20) enlisted as military pharmacist. He was sent to Waarden in Holland; the locality, surrounded by the enemy, owed to his ingenuity as chemist its escape from deprivation of drinking water and soap. After Waterloo (June 18, 1815) the siege was lifted and Caventou returned to Paris to resume his studies. In order to earn some money, he issued, through the generosity of a publisher, Mequignon-Marvis, a "Nouvelle Nomenclature Chimique" (1816). This book of 300 pages was a kind of dictionary, in which the author (then only 21) connected the new names with the old ones. For instance, carbon dioxide was formerly called gas sylvestre, sylvester spirit, fixed air, aërial acid, mephitic air, atmospheric acid, acid of chalk, acid of charcoal. Consequently, this table of synonyms made it easier to read the early writers. It went through several editions, a


NOUVELLE

NOMENCLATURE

CHIMIQUE,

PAR J. B. CAVENTOU,

A PARIS,

1816.

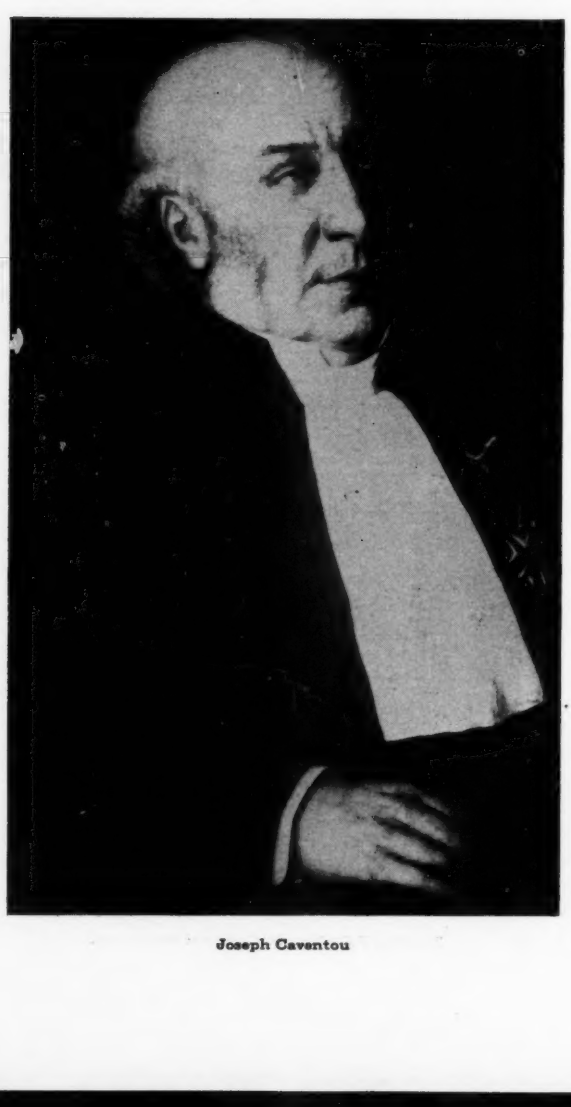
Title Page of Caventou's "Nouvelle Nomenclature Chimique"

L. G. L. Publicite, Paris

The New Pelletier and Caventou Monument

proof of its usefulness. However, the immediate monetary return was slight and after several months of privation he again was a candidate for the interneship. He succeeded, of course, and for the time being his needs were taken care of. His interneship brought him to the attention of the head men of the profession whose favor he quickly captured by his intelligence and industry. Moreover, he had at his disposal a laboratory in which he devoted himself to researches in organic chemistry. These brought him into contact with Pelletier, who by then was established on the Rue Jacob. Thus began an association which lasted until Pelletier's death. At the time of their first joint study (1817), Pelletier was 29 and Caventou 22.

From this time on, Caventou's university career was firmly established. Like Pelletier, he passed through the various academic grades with success. In 1820 he was elected to the Académie de Médecine; his father was made *correspondent* the next year. Because of his mastery of proximate analysis he was authorized in 1826 to deliver a course of special lectures in organic chemistry; this was the first course in organic chemistry given in Paris. In 1830 he became *adjoint* (assistant) professor and in 1835 there was finally created for him a chair of toxicology, which he occupied for 25 years. He retired voluntarily in 1860. Though he suffered a severe cerebral hemorrhage in 1874, he lived three years longer and died at the age of 82 on May 5, 1877. Like Pelletier he conducted a pharmacy; his was located on the Rue Gaillon.[1]



Joseph Caventou

Pelletier began his researches quite soon after he received his pharmacist licence (1809). He worked in his father's laboratory, which had already been made famous by Rouelle and Darcet the elder. He published several notes, among which particular interest attached to the studies which led him to his doctoral dissertation, namely, some persevering studies on a goodly number of gum resins: sagapenum, asafetida, opoponax, bdellium, myrrh, gum caragna, frankincense, etc. These studies were definitely conducted along lines quite different from those used by the early chemists, who employed almost exclusively destructive distillations, which yielded oils, empyreuma, volatile spirits, etc., and a residue (*catput mortuum*) characterized by its saline, tartarous, or caustic state. Pelletier, in contrast, applied successive solvents and so obtained different fractions. For example, opoponax yielded as many as ten different principles. Examination of the six papers which appeared in Volumes III, IV, and V of the *Bulletin de Pharmacie*, gives an idea of this type of investigation. Combined into a thesis (25 pages), these short accounts yielded a document which was accepted for his Docteur ès Sciences degree, which in those days was seemingly hedged about by a minimum of requirements.

As a student of Haüy, Pelletier presented a second thesis, known as the dissertation, which dealt with the value of physical characteristics used in mineralogy. In particular, he pointed out that an advantage can be gained from electrical conductivity.

Quite soon after his thesis was accepted, Pelletier published the following: a study of the compounds of gallic acid with vegetable materials (*3*); the analysis of sarcocolla (*4*); the chemical examination of several coloring matters derived from red sandalwood and alkanet (*5*); from turmeric (with Vogel) (*6*); and from olive gum (*7*). With this latter study he initiated the extraction of crystalline proximate principles. He gave the name *olivile* to a product obtained in this instance.

This brings the record up to 1817, when the first joint publication with Caventou appeared. It described the action of nitric acid on the nacreous material of human biliary calculi (cholesterol), and the new acid which results (*8*). There followed several other papers on croton oil, from which they separated crotonic acid (*9*); on ambergris, from which they isolated *ambreine*, an unsaponifiable substance resembling but not identical with cholesterol (*10*); on the coloring matter of cochineal, carmaine (*11*); on the pigment of

[1] Caventou's son Eugène was also a chemist; he was made a member of the Académie de Médecine during his father's lifetime.

leaves, chlorophyll (*12*); and later, in association with François, on the bitter principle of the cahinca root, which is due to the crystalline cahincic acid (*13*).

These scientists now embarked on the investigations in which they were going to display their real talents, namely, the field of the alkaloids. To gain a real appreciation of their work, it must be kept in mind that these studies were made 130 years ago. Scheele, likewise a pharmacist, had developed the method for isolating a number of vegetable acids, but these products had only a slight importance in comparison with the therapeutic properties of the plants from which they were obtained. In contrast, Derosne, Seguin, and then especially Sertürner, arrived progressively at the isolation of morphine. Sertürner (*14, 15*) announced that he had found "a new alkaline base, morphine, a very singular substance, which resembles ammonia, and which sheds some light on the other salifiable bases." Regarding its constituents, he wrote: "I have not had time to determine them; they are probably oxygen, carbon, hydrogen, perhaps also nitrogen." It is interesting to note that the presence of nitrogen did not make more of an impression. The existence of salifiable organic bases combining in a fixed proportion with mineral acids is so obviously comparable to the behavior of organic acids which, in a reverse sense, combine with mineral bases. This mental blindness extended even further. When Pelletier and Magendie obtained emetine from ipecacuanha (*16*) they stated that this substance yielded no ammonia when distilled, and consequently nitrogen did not enter into its composition. Later, at the discovery of strychnine in 1819, Pelletier and Caventou (*17*) again flatly declared regarding this base: "Distilled with copper oxide, it yields much carbonic acid and gives only traces of nitrogen, which we believe should be attributed to some particles of atmospheric air. Hence it is composed of oxygen, hydrogen, and carbon, and nitrogen does not seem to be one of its constituents." The same observations were later reported for brucine (*18*) and cinchonine (*19*). Bussy, who was then *préparateur* at the École de Pharmacie, was the first to prove the presence of nitrogen in morphine and to determine its quantity (*20*). His work is characterized by extraordinary attention to details and it is therefore recommended to beginners in organic chemistry, who need to learn and exercise the essential qualities of patience, skill, and precision. The word "precision" is not exaggerated here, since Bussy's figure, 4.5 per cent, agrees almost exactly with the theoretical nitrogen content, 4.6 per cent. This feat undeniably ranks as a prime discovery.

After this convincing piece of research it followed as a matter of fact that Pelletier could no longer maintain his opinion that these bases were nonnitrogenous. In collaboration with his friend J. B. Dumas, he therefore, in 1823, repeated his ultimate analyses, applying all the necessary refinements (*21*). This time quinine, cinchonine, brucine, strychnine, emetine, morphine, narcotine, and caffeine were all recognized to contain

Joseph Pelletier

nitrogen and in significant amounts. For instance, quinine was found to contain 8.45 per cent (theoretical value, 8.64 per cent).

To account for the inadequacy of the determinations by Pelletier and Caventou, Dumas and Pelletier stated in their paper: "It doubtless is surprising that the existence of nitrogen in the vegetable alkalies escaped Messrs. Pelletier and Caventou, as well as several other chemists who like they were occupied with the examination of these substances. . . . It seems, in fact, that this point is readily proved, but if we keep in mind the small amounts of the substances which they had at their disposal for this purpose in their first studies, it is easy to understand why the distillation in ordinary apparatus did not furnish them with very appreciable quantities of ammonia. . . . On the other hand, in the combustion with copper oxide, the ratio of carbonic acid to nitrogen is usually of a quite different order, which led those who observed this fact to ascribe it to the presence of atmospheric air in the tubes."[2]

Very much later, in 1832, Pelletier (*23*) analyzed aricine, piperine, ambreine, anchusic acid, santaline, olivile, etc. This time only the first two were found to contain nitrogen; the others did not.

Pelletier's first study of alkaloids was made in col-

[2] Actually, it is easy to detect the presence of nitrogen in several minutes with as little as 1 mg. of alkaloid (or even less). See (*22*).

laboration with François Magendie (1783–1855), the noted physiologist. They (*24*) found ipecacas to contain a particular substance, emetine, which was salifiable, showed alkaline properties, and clearly had the vomitive properties of ipecac. As in the case of morphine, it was possible here to find in a definite product the dominant qualities of the crude drug.

Two years later, in 1819, Pelletier and Caventou started their beautiful researches on the vegetable alkaloids by studying some strychnine plants (*25*). It is interesting to trace out one of their reasons for taking this direction. If one considers the members of a plant family, which through previous observations have been shown to possess the same medical virtues, it is likely that they contain the same proximate principles, whose intensity of action should be proportional to the amount present, or whose activity is due to one of them which has some well characterized dominant property. That is why they turned their attention to the Saint Ignatius bean (*Strychnos ignatii*) and to nux vomica (*Strychnos nux vomica*), which were both known to have marked tetanic effects. Having found the same alkaloid in these preparations they sought and found it also in snake wood (*Strychnos colubrina*), thus confirming the opinion of those botanists who regarded this plant as a strychnos. Pelletier and Caventou initially named this active vegetable principle *vauqueline*, in honor of the eminent chemist L. N. Vauquelin (1763–1829), but the commissioners of the Académie des Sciences thought "that a highly respected name ought not to be applied to a harmful principle." Consequently, the name *strychnine* was selected, indicating the botanical origin of the material. This latter name, in its turn, was criticized by Magendie, who preferred *tetanine* in view of its marked tetanic effects, just as *morphine* and *emetine* reflect the physiological characteristics of these substances. Subsequently, Pelletier and Caventou (*26*) discovered strychnine in *Strychnos tieuté* (*Upas tieuté*), but they found none in *Anthiaris toxicaria*.

It should be pointed out that they took pains to demonstrate that the toxicity of the strychnine from various botanical origins was identical. They expected to find strychnine also in the bark of the false angostura (*Brucea anti-dysenterica*), but instead they discovered that it contained a new alkaloid, namely, brucine (*27*).

Finally, with respect to strychnine, attention should be given to their paper on the methods used to extract this alkaloid (*28*). It was written to answer a colleague who claimed that he had not had to start with St. Ignatius's bean or nux vomica. The discordance arose from the fact that sometimes the brucine is very abundant.

Their celebrated work on cinchona is entitled, "Recherches chimiques sur les quinquinas" (*29*). It has several subdivisions: (*a*) Examen chimique de quinquina gris (*Cinchona condaminea*), (*b*) Examen chimique de quinquina jaune (*Cinchona cordifolia*), (*c*) Examen chimique de quinquina rouge (*Cinchona oblongifolia*).

With regard to (*a*), *i. e.*, gray cinchona, they tried particularly to repeat the work of Gomes, a Portuguese scientist, who had extracted from it a crystallizable substance called *cinchonin*, a name which was changed into *cinchonine*, since the ending *ine* had by this time been adopted for salifiable organic bases.

To obtain their cinchonine Pelletier and Caventou prepared an extract of gray cinchona and exhausted it with alkaline water. The insoluble portion contaminated with resin and fat was then treated with very dilute acid. The acid solution, after concentration, was mixed with an excess of very pure magnesia at a moderate temperature. After cooling the magma was washed with water, dried on the water bath, and then treated with alcohol. The alcoholic liquor on evaporation yielded a crystalline mass of cinchonine, which could then be purified.

It was by repeating this process, almost exactly, with yellow cinchona bark (*b*) that Pelletier and Caventou discovered quinine. There was this fundamental difference: instead of crystals, a transparent (yellowish) noncrystalline mass, much more soluble in ether than cinchonine, but, like the latter, producing well-crystallizing salts, was obtained.

The study of red cinchona bark (*c*) yielded both of the alkaloids at the same time.

At the close of their paper Pelletier and Caventou

Analyse des Observations de M. Double, *extrait de la* Revue Medicale, 6e. livraison, 1820;

Par M. Rouzet.

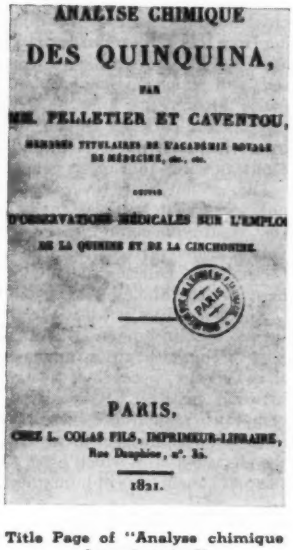
C'est dans les derniers jours de septembre et dans le courant d'octobre 1820, que M. Double a fait ses essais. On était alors, par rapport aux maladies régnantes, à la fin d'une catastase de fièvres intermittentes de divers types.

Première observation. — A la suite d'un séjour assez court dans la vallée de Palaiseaux (5 lieues nord-ouest de Paris) où les fièvres intermittentes étaient très-communes, la fille de chambre de M. D***, occupant une place importante dans l'université, contracta une fièvre intermittente tierce. A son arrivée à Paris, elle avait déjà eu trois accès bien complets, dont chacun avait duré de dix à douze heures. La malade n'éprouvait aucune douleur interne, ni pendant l'accès, ni hors de sa durée; il n'y avait point de symptômes d'irritation ni de gastricité. M. Double pensa que c'était le cas d'employer le nouvel alcali du quinquina. Il prescrivit donc neuf grains de sulfate de quinine, comme représentant la valeur environ d'une once de quinquina, à prendre en trois doses de trois grains chacune dans l'intervalle d'un accès à l'autre. L'accès suivant n'eut point lieu. Le lendemain et les deux jours suivans, le malade prit, matin et soir, une dose de quinine de quatre grains. Les six jours qui suivirent, elle continua la dose de quatre grains, mais le matin seulement. La fièvre n'a point reparu.

An Early Report on the Use of Quinine

It is interesting to note the incidence of fever in the Paris area.

gave a veritable dissertation on the usefulness of the extraction of proximate principles. By reason of the facts acquired through the employment of the whole drug (or of its crude extracts), there may be a tendency to question the possibility of replacing a drug by one of its proximate principles. If it is admitted that the associations such as are presented by plants are essential to their therapeutic action, it will be necessary "to ban the chemical sciences from the sanctuary of medicine." Pelletier and Caventou stated that experience ought to decide this matter, but from this time on they believed it "a fortunate circumstance that one could now administer the pure active principle in order to have it at its full power. Sometimes a patient cannot take an ounce of powder or a glass of liquid.... Moreover," they concluded, "we hope that some able practitioner, combining prudence with sagacity, will carry out therapeutic studies on the alkalies of cinchona, and thus give a medical utility to our work."


ANALYSE CHIMIQUE
DES QUINQUINA,
PAR
MM. PELLETIER ET CAVENTOU,
PARIS,
CHEZ L. COLAS FILS, IMPRIMEUR-LIBRAIRE,
1821.

Title Page of "Analyse chimique des quinquina"

Soon after the preparation of quinine sulfate was accomplished, Pelletier and Caventou published (1821), a book of 88 pages, entitled, "Analyse chimique des quinquina," followed by "d'Observations sur l'emploi de la quinine et de la cinchonine." This brochure contained a methodical examination of the principal pharmaceutical preparations which had cinchona as a base. Among the physicians who volunteered to give quinine a fair trial were Double, Fouquier, Chomel, Coutanceau, Magendie, Devèze, etc. The results were unequivocal. The patients' fevers were arrested and it was proved that quinine could advantageously take the place of the various powders and other nauseating cinchona preparations.

This publication was supplemented by chemical studies of gray, yellow, and red cinchona, and by analyses of other drugs called cinchonas. Papers were published by Pelletier alone on cinchona from Cartagena (*Portlandia hexandra*) which contains cinchonine and quinine; cinchona from Ste. Lucie or cinchona-Piton (*Cinchona floribunda*), which contains none (*30*); and quinanova, which contains only a particular acid, namely, quinovic acid (*31*).

In 1825 Pelletier, together with Petroz, studied a bark known as *Quina bicolorata;* neither quinine or cinchonine was found (*32*).

This same year Pelletier was able to announce (*33*) that he had finally obtained quinine itself in crystalline form. If the fact of obtaining a number of salts having a well-defined crystalline form put the chemists' conscience at rest "his mind could still not be fully satisfied," and therefore he was determined to obtain the crystallized base. He succeeded in this endeavor by dissolving the compound in 40–42 per cent alcohol, and allowing the solution to stand in a cold place.

Several years after the discovery of quinine, Pelletier and Caventou (*34*) addressed a letter to the members of the Académie des Sciences regarding the manufacture of quinine. This communication was prompted by their candidacy for the prize established by Baron Monthyon. Their letter is interesting because it shows the development of their discovery. With admirable disinterestedness, they described the method of preparation, whose principles were briefly outlined above. At first they made the quinine sulfate themselves, but as the demand increased, Pelletier, Robiquet, and Levaillant carried out this procedure on a large scale in their factories, and later they arranged for still other manufacturers to engage in this operation. Pelletier, Levaillant, and Delondre (associated with Pelletier) had each treated, in 1826, 159, 300 kg. of cinchona, which yielded an average of 23 g. of quinine sulfate per kg.

The authors of this letter pointed out that if there resulted an unlooked for market for the manufacturers, this industrial development should also be regarded as adding to the renown of France, and that, on the other hand, if, by reason of the fortunate applications of quinine sulfate, it could in 1820 be considered an important chemical discovery, it might be regarded as a capital advance in medicine.

The Monthyon prize of 10,000 francs (equals 2 million in 1950) was awarded by the Académie des Sciences to Pelletier and Caventou at its meeting on June 11, 1827. It is interesting to note the importance attached to quinine by the Académie, since at the same time it also conferred a medal of encouragement worth 2000 francs on Ossian Henri for having perfected the art of extracting quinine sulfate and for having greatly brought down the commercial price of this salt (*35*).

However, the use of this product did not spread as rapidly as the initial success had promised. Broussais, among others, feared the side effects on the digestive organs, and the respect for his authority was such that the military physicians—at the annexation of Algeria—hesitated to use this remedy, even though the army was being riddled by malaria to such an extent that the members of the legislative body proposed the abandonment of the occupation. Fortunately, a young army doctor, Maillot, did not hesitate to prescribe quinine sulfate. The finest reward which the soldiers could give to Dr. Maillot was certainly to refer to his as "the service where one no longer dies." Indirectly, this praise also honored the chemists responsible for the discovery of quinine, and equally it served to refute the pontifical and stubborn authority of Broussais.

The consideration of botanical families led Pelletier

and Caventou to a similar study of three colchicia: cevadilla (*Veratrum sabadilla*); white hellebore (*V. album*); and a meadow saffron or autumn crocus (*Colchicum autumnale*). This paper (*36*) appeared the same year as the study of cinchonas. They believed that all these plants contained veratrine, but it is now known that the alkaloid of the colchicum is not veratrine. This work preceded even the famous paper on the cinchonas.

In some instances, Pelletier published important studies alone. For example, black pepper (*Piper nigrum*) gave him a particular neutral crystalline material (*37*) (previously suspected by Oerstedt), which Pelletier named piperine or piperin. (This substance is now known to be an amide.)

He announced the discovery of an organic alkaline principle, different from picrotoxine, in cocculus indicus (*38*). This study was repeated with J. P. Coverbe; they discovered menispermine and paremenispermine (*39*). Under these circumstances, they stressed the fact that although not an alkaloid, picrotoxine represented the active principle of cocculus indicus.

Together with Coriol, Pelletier discovered a new base, which he named aricine (*40*). This was found in a bark coming from Africa or Peru, and exported as yellow cinchona. With Petroz, he took up the study of curare (*41*). Finally, although opium had already been the subject of numerous investigations, Pelletier succeeded, through nicely executed manipulations, in adding a new alkaloid to the list of those already known, namely, narceine (*42*).

In a study of the products derived from the treatment of resin (pine turpentine) for lighting by gas, Pelletier, in association with Ph. Welter (*43*), characterized, under the name *retinaphte*, a fraction, C_7H_8, whose theoretical relationship with the benzene compounds was clearly emphasized. This material was subsequently called toluene. With the same collaborator, he also studied the distillation of bitumens. Numerous liquid fractions were collected and analyzed; they were found to consist of hydrocarbons (*44*).

Two papers were published by his collaborators after Pelletier's death. He and Welter had studied the oily products which accompany the succinic acid when amber is destructively distilled (*45*). These are hydrocarbons boiling from 130° up to 400°; their carbon content runs from 88.6 to 90.5 per cent. Finally, with H. Deville (*46*), he distilled guaiac resin; among the products was an oil, $C_7H_8O_2$, which is nothing other than guaiacol.

From all that has been outlined above, it is apparent that Pelletier confined himself almost entirely to the organic field. However, one important paper of another kind should be noted. In this, "Facts relative to the history of gold," he showed that gold peroxide (tervalent Au) combines with bases (*47*), and also made numerous other judicious observations.

Some papers of minor importance which were published by Pelletier or Caventou will not be discussed here. It will only be pointed out that these two scientists were so modest and lacking in self-interest that their contemporaries admired them for these qualities. This attitude is well illustrated by the words of J. B. Dumas, who in his funeral oration for Pelletier said:

> Pelletier made a place for himself in science which cannot grow less. His discoveries are such that they cannot be effaced or diminished, because they are absolute discoveries. He discovered new compounds; he has given science some hitherto unknown substances, and as long as chemistry itself survives in the memory of men, the name of Pelletier will be spoken with respect and gratitude.
>
> How can it be otherwise, when we see this name, associated with one who shared his highest glories, when we see this name which is linked with the greatest discovery of modern therapeutics: sulfate of quinine.
>
> The thing of which Paracelsus and his disciples dreamed, this great art of extracting the quintessence from plants, of reducing large masses of repulsive pharmaceutical products to a hardly appreciable volume, this was the goal which Pelletier set for himself, and which he succeeded in reaching in a large number of cases, but never, it must be confessed, in a manner as fortunate or complete as when he accomplished the extraction of quinine from cinchona, in a celebrated research which established his reputation and that of his collaborator Caventou.
>
> Did we not hear, several years ago, when the Académie had not yet added Pelletier to its membership, the spokesman of the Monthyon commission solicit and obtain by a unanimous vote a grand prize as reward for this discovery by proclaiming the name of Pelletier and that of Caventou as worthy of a permanent place among the benefactors of humanity?
>
> And when it is recalled that the discoverers of quinine sulfate turned over to humanity in its entirety a discovery which could have brought them an immense fortune, and when it is known that Pelletier, because of this generosity, went through a period in which his own patrimony was endangered by an ungrateful competition, there are revealed in the beauty of this discovery, in the philosophic sentiment which presided at its publication, all the characteristics which justify in fact the placing of him among the real benefactors of humanity.

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(30) *J. Pharmacie*, [2] **7**, 101, 114, 302 (1821).
(31) *Ibid.*, [2] **7**, 109 (1821).
(32) *Ibid.*, [2] **11**, 449 (1825).
(33) *Ibid.*, [2] **11**, 249 (1825).
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THE SEPARATION OF ANIONS BY PAPER PARTITION CHROMATOGRAPHY[1]

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A PROCEDURE has been described by Frierson[4] for the separation of cations by paper partition chromatography. This procedure has been adapted to the separation of a group of anions consisting of phosphate, arsenate, sulfide, ferricyanide, ferrocyanide, and iodide ions. These ions were chosen because they are common ones that give colored precipitates with silver ion.

The separation was carried out in a can about 50 cm. tall and 15 cm. in diameter. The can was covered with a piece of cardboard during use. A graduated cylinder of suitable height was placed inside the can to serve as a support for a Petri dish which held the developer. A mixture of 40 per cent of *n*-butanol, 40 per cent of ethanol (95 per cent), and 20 per cent of water by volume was used as the developer.

A strip of filter paper 25 cm. long and about 2 cm. wide was notched on each side about 1 to 2 cm. from one end. This notching narrowed the width of the paper at that point and so restricted the flow of the developer. The solution of the ions, which was 0.5 *M* with respect to each anion, was put on the paper strip about 1 to 2 cm. from the notch on the long side by means of a dropper that had been drawn out to a fine point. The strip of paper was placed on a blotter and the dropper containing the solution was drawn quickly across the strip to form a line straight across and about 0.1 cm. in width. Too wide a line caused the bands to be too wide and not well defined. The line should be uniform in width. There was a tendency for a wide spot to form at the edge where the dropper first touched the strip. This was overcome by pressing the strip firmly against the blotter. The strip of paper was then folded between the notch and the line of solution and the short end hung over the edge of the Petrie dish and dipped into the developer. The long end of the strip then hung, without touching anything, in the can. As many strips could be developed at once as the dish would hold without crowding. After the strips were placed, the can was covered and allowed to stand for 20 to 24 hours at a temperature of 20° to 25°C. During this time the anions were moved down the strip and separated. The strip was then removed, dried with a hot air blast, and sprayed with 0.1 *M* silver nitrate solution from an atomizer. Bands of colored precipitates formed to show the locations of the anions. A blank was run to detect any bands due to impurities. None was found.

The order of separation, from top to bottom, was: ferrocyanide, ferricyanide, sulfide, arsenate, phosphate, and iodide. The ferrocyanide appeared on spraying with silver nitrate solution as a 2-mm. light green band with a light yellow band below and adjoining it. The ferricyanide appeared as a 2-mm. orange band just below the ferrocyanide. The sulfide appeared as a 5-mm. gray-brown band. The arsenate appeared as a 1-cm. red-brown band. The phosphate appeared as a very light 1-cm. band that darkened fast on exposure to light of the room. The iodide appeared as a 1-cm. yellow band that darkened slowly on exposure to light of the room. The approximate Rf values of these ions at 30°C. are: ferrocyanide, 0.12; ferricyanide, 0.15; sulfide, 0.16; arsenate, 0.26; phosphate, 0.37; and iodide, 0.92. Temperature change caused considerable variation in the rate of movement of the ions but did not change the order.

[1] This paper is based on work done by Charles Drinkard in a senior course in Independent Study.

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[4] FRIERSON, W. JOE, AND MARY JO AMMONS, THIS JOURNAL, 27, 37–8 (1950).

A GRAPHICAL FREE ENERGY APPROACH TO UNARY SYSTEMS

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It is well known that the significance of many important thermodynamic relations is more likely to be appreciated if they are interpreted graphically, and it occurred to the writer that the concept of free energy could be so interpreted to the student's benefit. Experience shows that equations such as the variation of free energy with pressure or with temperature will not be regarded as mere mathematics if one utilizes the following simplified treatment of Gibbs' thermodynamic surfaces,[1] particularly if the treatment is correlated with the familiar unary or one-component phase diagrams, as shown below, thereby making the free energy function more vivid.

The equation relating infinitesimal changes in the free energy (F), pressure (P), and absolute temperature (T) of a closed system of volume V, and entropy S, in equilibrium with its surroundings, namely,

$$dF = VdP - SdT, \qquad (1)$$

may be used as the starting point. While this relation is valid for *any* such closed system it is convenient here to confine its application to one-phase unary systems. With these in mind the variation of the free energy with pressure at constant temperature is given accordingly by $(\partial F/\partial P)_T = V$. Free energy must increase, therefore, with pressure, at constant temperature, because V is always positive. It follows by differentiation that $(\partial^2 F/\partial P^2)_T = (\partial V/\partial P)_T$, which is, however, always negative by everyday experience. We see, then, that if F be plotted as ordinate against P as abscissa for a closed system at a given temperature a curve will result having a positive slope, the slope decreasing with increasing pressure. Furthermore, since $(\partial V/\partial P)_T$ decreases with increased pressure (for compressibility decreases with rise of pressure) the above-mentioned slope will decrease with rise of pressure more slowly at higher pressures than at lower. This leads us to the general isothermal free energy curve in Figure 1, the slope approaching zero as pressure is increased indefinitely. The absolute values of the ordinates are, of course, unknown.

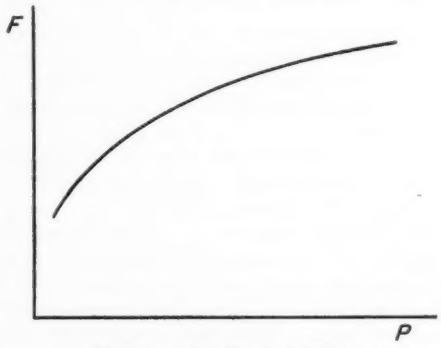

Figure 1. Free Energy Isotherm

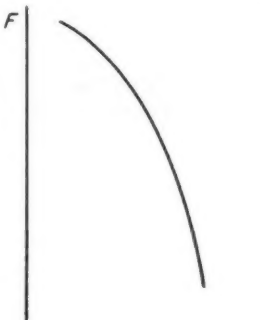

Figure 2. Free Energy Isobar

The variation of free energy with temperature at constant pressure, which also follows from equation (1), namely, $(\partial F/\partial T)_P = -S$, may be treated graphically in a similar manner. Entropy is regarded as a positive quantity and therefore a plot of F versus T for a given pressure will have a negative slope. This slope also decreases *algebraically* with rise in temperature because $(\partial^2 F/\partial T^2)_P = -(\partial S/\partial T)_P = -C_P/T$ where C_P is the heat capacity at constant pressure. The slope, however, is negative, so that its *numerical* value *increases* with rise in temperature. Moreover, since T increases more rapidly than C_P with rise in temperature, the slope changes more slowly at higher than at lower temperatures. These facts are represented in the isobaric free energy curve of Figure 2.

These variations of free energy with pressure and temperature may now be embodied in a three-dimensional plot, shown schematically in Figure 3, to give a curved free energy surface which is convex from above. Clearly, any horizontal section of the surface corresponding to a fixed value of F will give a curve with slope given by $(\partial P/\partial T)_F = S/V$ as required by equation (1). It may be remarked here that whether we regard absolute free energy values as being positive or negative is a matter of convention. In the accompanying figures positive values have been implied but the essentials of the present discussion are independent of the convention chosen.

The above considerations have been confined, as already stated, to any one-phase, one-component system, whether solid, liquid, or vapor, and any one of these three will have a free energy surface such as that of

[1] Gibbs, J. W., "Collected Works," Longmans, Green, and Co., Inc., New York, 1928.

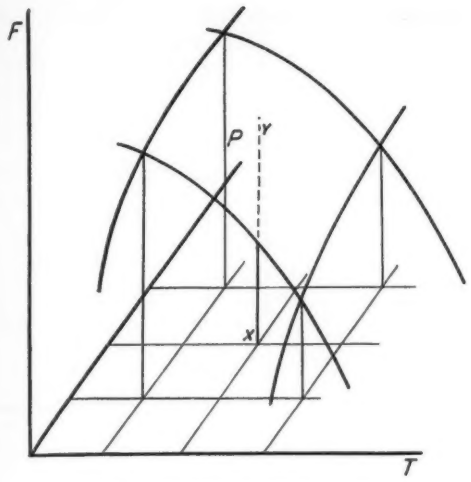

Figure 3. Free Energy Surface

Figure 3, although only a portion of that surface may be realizable experimentally because of the limited range of temperature and pressure over which that particular phase is stable. The range of conditions for stability will be considered below.

Another feature of a plot such as Figure 3 was pointed out by Gibbs.[2] If at the point y a tangent plane to the surface be drawn the intercept of this plane on the F axis gives the value of the internal energy of the phase at the pressure and temperature of y. This may be shown by reference to Figure 4 as follows:

Let y be the point of contact of the tangent plane yBCD. Now

$$\begin{aligned}\overline{OC} &= \overline{QJ} = \overline{QG} - \overline{JG} = \overline{QG} - (\overline{JD} - \overline{GD}) \\ &= \overline{QG} - \overline{JD} + \overline{GD} \\ &= \overline{QG} - (\partial F/\partial P)_T(\overline{CJ}) + (\partial F/\partial T)_P(\overline{yG}) \\ &= F - VP + (-S)(-T) = F - PV + TS\end{aligned}$$

where F, V, P, S, and T refer to point y. But $F + TS = H$, the heat content. Therefore $\overline{OC} = H - PV = E$, the internal energy.

We may now correlate the above discussion with unary phase diagrams. Imagine a unary system which forms only one solid and one liquid, and, of course, only one gas phase. Each of these three will have its own F surface which, when plotted on the same set of coordinate axes and for the same quantity of substance (one mol, for example) will intersect the other two. This is because, at any given temperature and pressure, the slopes are different, for the molar volumes and entropies are different in the three states. Any vertical line, such as xy of Figure 3, corresponding to a given temperature and pressure, will cut each surface at a point representing the free energy of the respective states, and, since in a unary system that phase with the lowest molar free energy is the stable one, the first surface to be cut by the line xy as it is produced upward gives the stable phase. Furthermore, since any two such surfaces will intersect along a space curve the latter gives the values of F, P, and T for the coexistence of the phases for such surfaces.

This will be made clearer by Figure 5 which shows two F surfaces intersecting along the space curve ab. *For the temperature and pressure* of point M, since MF' is less than MF'', the state of aggregation corresponding to the surface on which F' lies is more stable than that

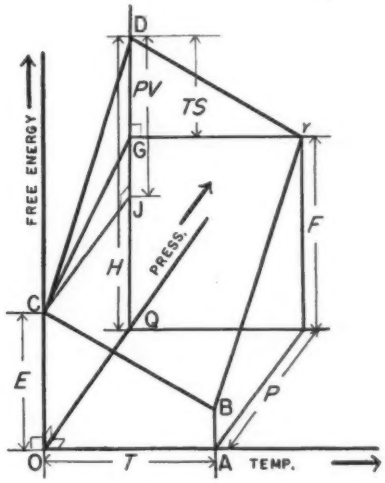

Figure 4.

for F''. It may be noted that the situation is reversed for point N. If, now, we visualize the upper surface as one for vapor and the lower as one for liquid, the line ab gives the conditions of F, P, and T for their coexistence, and the projection of ab on the P-T plane is nothing more than the familiar vapor pressure curve of the liquid. As such the relation between P and T should be

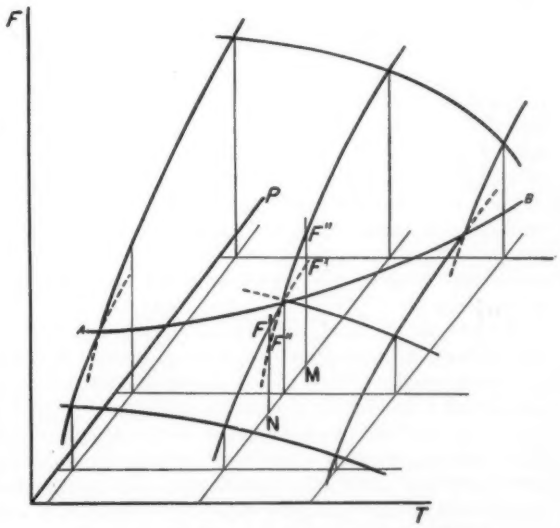

Figure 5. Conditions for Coexistence of Two Phases

[2] Gibbs, J. W., *loc. cit.*, Vol. 1, p. 116.

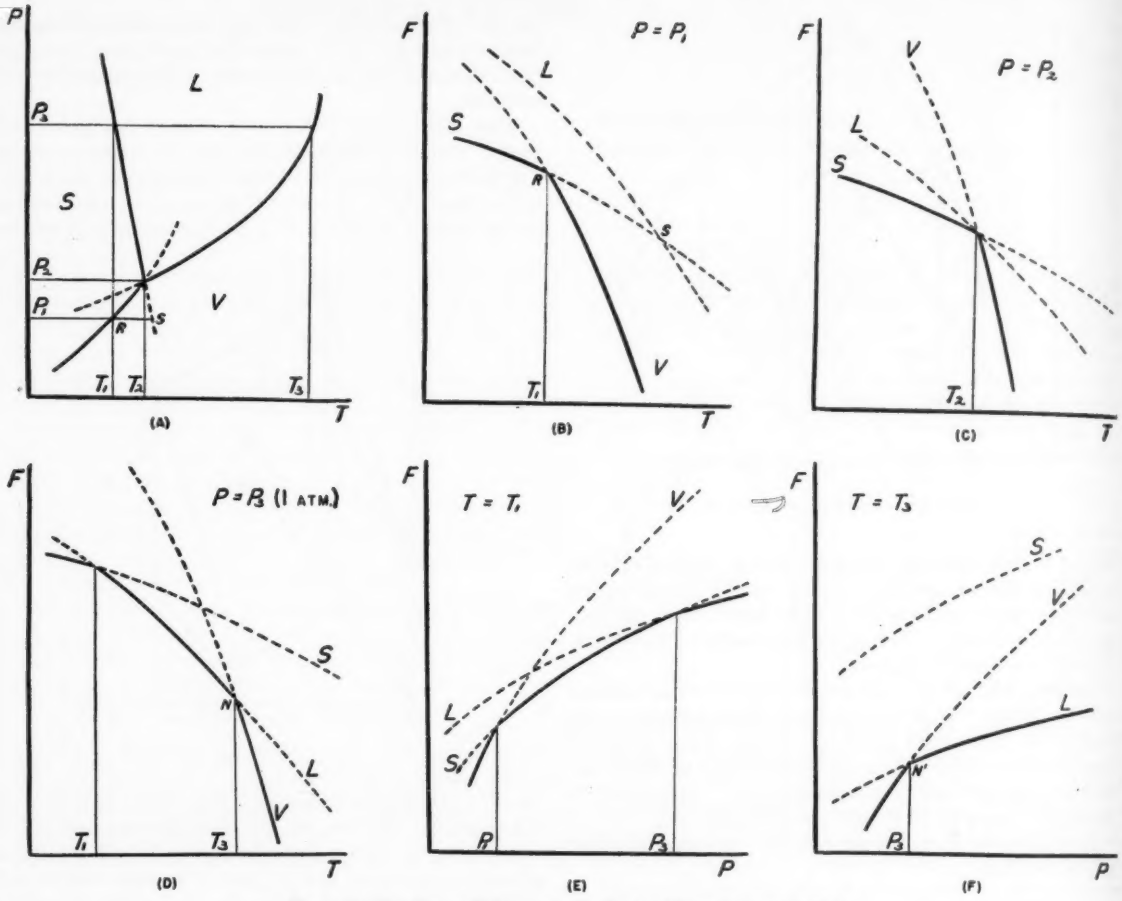

Figure 6. Free Energy Relations in the System Water (schematically)

given by the Clapeyron equation, and this may be shown immediately by writing equation (1) for each surface and equating dF_{vapor} and dF_{liquid} (since $F_{\text{vapor}} = F_{\text{liquid}}$ on *ab*), yielding $dP/dT = \Delta S/\Delta V$, where $\Delta S = S_{\text{vapor}} - S_{\text{liquid}}$ and $\Delta V = V_{\text{vapor}} - V_{\text{liquid}}$. It may be pointed out, too, that as one passes from *a* to *b* the values of *F* decrease, for, with liquid-vapor equilibria, the increasing effect on *F* of increased pressure is more than offset by the decreasing effect of increased temperature.

The foregoing arguments can obviously be extended readily to as many surfaces as there are phases of the substance. Four surfaces, for example, would be required for a substance which exhibits two allotropic solid forms.

In order to avoid visualizing the intersection of several surfaces in three dimensions we may draw isobaric and isothermal sections of the *F-P-T* model. This is illustrated as follows for the substance water. Figure 6 (*a*) is its familiar phase diagram (schematically) and Figures 6 (*b*), (*c*), and (*d*) typical isobaric sections for pressures P_1, P_2, and P_3, respectively. The sections of surfaces for solid, liquid, and vapor are labeled *S*, *L*, and *V*, respectively. For a given temperature on each isobar it will be noted that *numerically* the slope for vapor > slope for liquid > slope for solid, for this is the relative magnitude of the entropies. The unbroken portions of these curves, being those of least free energy for the given temperature and pressure, represent the stable phases, and the broken lines, therefore, metastable phases. (The reverse was true for Figure 5.) A point of intersection of two unbroken lines is a stable two-phase system, for example *r* in Figures 6 (*a*) and (*b*); point *s* is, correspondingly, a metastable equilibrium. If the pressure P_3 corresponds to one atmosphere it is evident that the temperatures T_1 and T_3 in Figure 6 (*d*) are 273° and 373°K., respectively. Figures 6 (*e*) and (*f*) are typical isothermal sections of the model for temperatures T_1 and T_3, respectively, of Figure 6 (*a*). For these, at a given temperature and pressure, the slope for vapor > slope for solid (ice) > slope for liquid, because this is the relative magnitude of the molar volumes. (Ice contracts on melting.) The interpretation of these isotherms and their relation to the phase diagram is apparent. For most substances, of course, the molar

volume for liquid is greater than that for solid at a given temperature and pressure so that the order of the slopes in the isotherms is different from that shown. The sketching of these and their correlation with the corresponding phase diagram is left as an exercise for the student. A similar assignment might be made of the system sulfur using the well-known phase diagram with three stable triple points.

The behavior at the critical point deserves special mention.[3] In unary systems critical phenomena are known only between liquid and vapor. Referring, then, only to the F surfaces of these two, if the F-T isobars such as Figure 6(d) were drawn for gradually increasing pressures we should find that the discontinuity in the slope at n, where the L and V curves intersect, would just disappear in the critical pressure isobar, making the two curves identical at all higher pressures. Similarly, if F-P isotherms such as Figure 6 (f) for gradually increasing temperatures were drawn, the discontinuity in the slope at n' would just disappear in the critical temperature isotherm. In terms of the F surfaces, ab in Figure 5 would come to an abrupt end at the critical point because the liquid and vapor surfaces have merged there to become a surface for fluid.

[3] *Cf.* J. C., SLATER, "Introduction to Chemical Physics," McGraw-Hill Book Co., Inc., New York, 1939, p. 174.

A THERMOREGULATOR FOR GAS-HEATED BATHS

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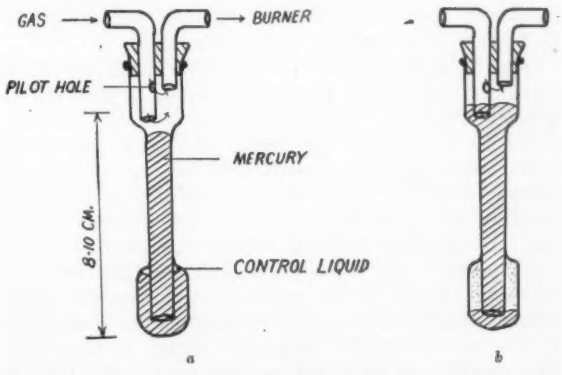
THIS regulator is designed to control the flow of gas to a Bunsen burner heating a constant-temperature bath which must have a moderate degree of regulation. It is based upon a mercury-valve action, controlled by the vaporization of a "control liquid" boiling at the temperature desired for the bath.

Operation is as follows: With the bath cooler than the desired temperature, the regulator is as in a, with gas flowing freely through it. When the temperature reaches the boiling point of the control liquid, this liquid vaporizes, forcing the mercury up into the upper chamber and cutting off the gas, as in b. A pilot hole in the inlet tube keeps the burner lighted.

Construction and dimensions are given in the figure. These dimensions are not critical, but a few design considerations should be borne in mind: (1) The volume of the control liquid must be enough so that its vapor will nearly fill the bulb when hot. (2) The bulb must be large enough so that the mercury forced out of it will rise far enough into the upper chamber to cut off the gas. (3) The stem may be any length, but it should be entirely inside the bath, in order to avoid heat conduction and cooling of the control liquid. Note also that the longer the mercury column, the higher the boiling point of the control liquid. (4) The pilot hole should be large enough to give a flame which will allow the bath to cool very slowly.

Advantages of this regulator are simplicity of construction, self-adjustment, and lack of need for calibration. It can be built by ordinary glassblowing techniques; any excess control liquid simply bubbles out the first time the regulator is used; and the control temperature is fixed by the boiling point of the liquid used.

Regulation is only moderate. It has been found easy to control a bath to within one degree with no special precautions. Probably it would do somewhat better than this with proper precautions.



No great amount of originality is claimed for this regulator. The author has seen other gadgets using the boiling-point principle, but no regulator like this.

A COURSE IN INSTRUMENTAL ANALYSIS

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THE past decade has witnessed a remarkable resurgence in interest in analytical chemistry. That "rare bird," the analytical chemist, has at last achieved recognition as an indispensable member of the research team which is used today to attack a complex problem of modern chemistry in any of its branches. The increasingly important role of the analytical chemist in industrial chemistry has been discussed by C. Rosenblum (*1*).

The increased prestige of the analytical chemist may be attributed, in part, to his considerable success in solving the difficult analytical problems which have been presented by the sudden demand for formerly seldom used or unknown materials such as the antibiotics, and uranium and the newer elements formed from it by transmutation. Much of this success has been achieved by the development of new physical methods of analysis. These methods, commonly called instrumental methods because of the necessary emphasis on instrumentation, have opened a new field to the analytical chemist, and have greatly broadened his vision.

The present widespread interest in instrumental methods is indicated by current analytical literature. A survey (*2*) for the year 1946 showed that 56 per cent of all research papers on the broad subject of chemical analysis dealt with instrumental methods. The annual reviews of analytical chemistry which appear in *Analytical Chemistry*, beginning with the year 1949, devote more than half of the total space to discussions of instrumental methods of analysis. G. Ewing (*3*) has recently described in THIS JOURNAL an undergraduate course in special methods of analysis involving the utilization of several instrumental methods.

The colleges and universities have been quick to recognize the need for providing training in this rapidly growing field, but they are confronted with difficult problems in fulfilling the need. It is not easy to fit new courses into an already crowded curriculum especially since it is recognized that training in the older, so-called wet methods, must not be abandoned. Then too, instrumental methods require instruments which are expensive, and which are so complex that they can rarely be made in the ordinary departmental shop. Educational institutions will require financial assistance if they are to supply adequate training in this field. The problem of financing modern instrumentation in academic institutions has been discussed editorially by W. J. Murphy (*4*). In spite of these difficulties academic training is now being provided. A survey of the teaching of instrumentation made in 1947 by the Fisher Scientific Company (*5*) showed that in 167 institutions more than one-third already were giving a specific, separate credit course in instrumentation. An additional one-third stated that they fully expected to offer such a course not later than the spring term of 1948.

Since 1946 the chemistry department of Rensselaer Polytechnic Institute has offered a four-semester-hour course in instrumental analysis. Each week two hours are spent in the classroom and two three-hour periods in the laboratory. The course is required for undergraduate chemists (approximately twenty students) in the first semester of the senior year, and is offered as an elective for chemical engineers or others who have the prerequisites of two semesters each of analytical, organic, and physical chemistry. A similar course is required of first-year graduate students who have not had equivalent training in their undergraduate work. It will be noted that the course is given at a time which will permit those interested to continue work in the field in the form of a research thesis. In addition it has been found that students doing a thesis in fields other than analytical chemistry have made use of the methods and techniques acquired during the course. Most of the research topics of the physical chemists, particularly in kinetics, require analytical measurements. The techniques of absorption spectroscopy are valuable to the synthetic organic chemist in determining the structures of the compounds that he has made. The experimental projects of the chemical engineer often require the use of analytical methods. Experience at Rensselaer has shown that once a student has had a course in instrumental analysis, he no longer approaches an instrument with fear and trembling, but rather fondly regards it as a possible solution to his particular problem.

A course in instrumental analysis can conveniently be divided into three parts, absorption spectroscopy (including fluorometry), emission spectroscopy, and electrometric procedures. Each of these fields is considered in turn below. The actual experiments, the equipment used, and the results obtained by students are discussed in some detail.

ABSORPTION SPECTROSCOPY

The first experiment is a qualitative study in the ultraviolet. A Beckman Model DU Spectrophotometer equipped with a hydrogen-lamp source is used. The student is given an unknown sample of a simple

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aromatic organic compound such as toluene or benzene, and some iso-octane purified by the conventional silica gel treatment (*6*). Using solutions approximately 1 per cent by volume of the aromatic compound in iso-octane, the ultraviolet spectrum is obtained from 200 to 400 millimicrons at wave-length intervals dependent upon the absorption peaks obtained. Then, using the American Petroleum Institute series of ultraviolet spectra, the student identifies his unknown.

The second experiment is a quantitative determination of manganese in steel by photometric measurements in the visible region of the spectrum. Each student is given an analyzed steel of manganese content unknown to him. Standard samples containing a half-gram sample of ingot iron and known amounts of a standard manganese solution are then prepared by the student. The unknown is treated along with these standards in 250-ml. pyrex volumetric flasks using the conventional treatment of solution in an acid mix consisting of equal parts of nitric acid, phosphoric acid, and water followed by oxidation with potassium periodate and subsequent dilution. The absorption spectra of one of the known samples and of the unknown solution are measured on both a Beckman Model B Spectrophotometer and a Coleman Model 10S Spectrophotometer from 350 to 1000 millimicrons. The wave length at which the maximum absorbance occurs is noted and appropriate filters are selected for the Klett-Summerson and Evelyn filter photometers. Calibration curves are prepared on all four instruments, and the per cent of manganese in the unknown sample is determined. Colorimetric measurements of the unknown, and an appropriate standard are also made with a Duboscq colorimeter. The use of several types of instruments (colorimeter, filter photometer, and spectrophotometer) enables the student to appraise the assets and defects of each type. Table 1 shows typical student results obtained on representative unknown samples on the various instruments mentioned. The deviation between the maximum and minimum values reported by the large number of students is surprisingly small for each of the instruments used except for the visual type Duboscq colorimeter.

TABLE 1
Manganese Results on Steel Samples

% Mn, Thorn Smith value	No. of student Determinations	Manganese, %				
		Duboscq	Klett-Summerson	Evelyn	Coleman	Beckman
0.28	10	Max. 0.30	0.30	0.29	0.30	0.29
		Min. 0.25	0.24	0.28	0.28	0.29
		Av. 0.28	0.28	0.29	0.29	0.29
0.42	10	Max. 0.48	0.43	0.42	0.46	0.44
		Min. 0.38	0.37	0.38	0.38	0.38
		Av. 0.42	0.41	0.41	0.42	0.42
0.61	10	Max. 0.78	0.64	0.64	0.63	0.63
		Min. 0.44	0.58	0.61	0.58	0.61
		Av. 0.62	0.62	0.62	0.61	0.62
0.74	10	Max. 0.82	0.78	0.77	0.77	0.78
		Min. 0.68	0.71	0.72	0.74	0.73
		Av. 0.74	0.73	0.74	0.75	0.75
0.80	5	Max. 1.01	0.80	0.81	0.84	0.84
		Min. 0.80	0.76	0.76	0.74	0.75
		Av. 0.86	0.78	0.79	0.79	0.81
0.86	9	Max. 0.96	0.90	0.91	0.91	0.92
		Min. 0.82	0.85	0.81	0.86	0.89
		Av. 0.89	0.89	0.89	0.89	0.91

In the third experiment, the student measures the absorption spectrum of an organic compound in the infrared region using a Perkin Elmer Model 12B Recording Infrared Spectrometer. The wave lengths of

Figure 1. Student Obtaining an Infrared Spectrum Using an Infrared Recording Spectrometer

the absorption bands are obtained from the prism calibration curves, and the student then identifies the functional group frequencies present by referring to tables such as those published by Barnes, Gore, Stafford, and Williams (*7*), Thompson (*8*), and Colthup (*9*). The organic compounds selected for the undergraduate student are ones which have several functional groups. In the case of the graduate student, particularly an organic major, the student is allowed to select his own sample since often he has several unknowns which he is anxious to study. Such a method as this reveals to the student not only the benefits but also the limitations of infrared spectroscopy as a qualitative tool.

The final experiment in absorption spectroscopy is a quantitative fluorometric determination. Fluorescence is caused by the absorption of ultraviolet light and emission of this energy in the form of visible light. As a practical application of fluorescence in instrumental analysis, a determination is made of aluminum in steel using a method developed in this laboratory. The instrument used is a Klett Fluorometer, Model 2070. Using a Vycor beaker, a one-gram sample of steel is dissolved in an acid mixture containing nitric acid, perchloric acid, and water. The sample is heated until fumes of perchloric acid appear, and then fumed for approximately five minutes, cooled, and diluted to 250 ml. An aliquot is selected dependent upon the suspected aluminum content, electrolyzed with a

Figure 2. A Student Obtaining an Ultraviolet Spectrum

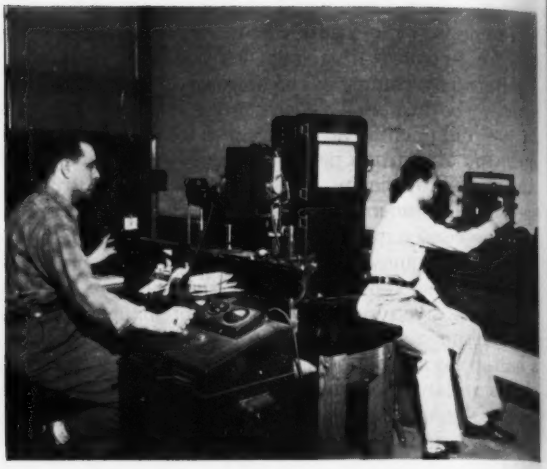

Figure 3. General View of the Emission Spectroscopy Laboratory

mercury cathode, and treated with 8-hydroxyquinoline and chloroform as described by Wiberley and Bassett (*10*). However, in place of the colorimetric measurement described in their procedure, a fluorometric measurement is made on the chloroform solution of the aluminum quinolate. This method has advantages over the colorimetric method in that a smaller sample may be taken for electrolysis, and small amounts of titanium and vanadium do not interfere. Some of the equipment used by the students in our absorption spectroscopy laboratory may be seen in Figures 1 and 2.

EMISSION SPECTROSCOPY

Qualitative Analysis. The instrument used is a Baird 3-meter Grating Spectrograph. The student is given an unknown brass sample and a sample of R. U. powder. R. U. powder is a standard sample first made by J. W. Ryde and H. C. Jenkins of the Research Laboratories of the General Electric Company, Wembley, England, and is marketed in this country through Jarrell-Ash Company in Boston, Massachusetts. It consists of a base mixture of calcium, magnesium, and zinc oxides to which the proper amounts of 47 other elements have been added in such amounts that only a few of the persistent lines (Raies Ultimes) appear for each element in the spectrum of the powder.

The spectra of the unknown brass sample and R. U. powder obtained with a d.-c. arc source and the spectrum of pure copper obtained with an a.-c. spark source are superimposed on an Eastman Kodak Spectrum No. 1 Analysis Plate. The student then identifies the elements present in his sample by comparing his plate with a plate which has an R. U. powder spectrum with most of the lines identified. This latter plate is prepared by the instructor. The student reports those elements with three or more lines shown on the plate as definitely being present in the unknown sample.

Quantitative Analysis. The same spectrograph as above is used together with a Leeds and Northrup densitometer. Figure 3 shows two students using this equipment. The samples used are aluminum samples which were obtained through the courtesy of the Aluminum Company of America. Each student measures three unknown samples and three standard aluminum samples using the conventional Petrey spark stand. Use is then made of the two-line calibration method as described by J. R. Churchill (*11*) to determine the percentages of the various elements present in the unknown samples. Table 2 shows typical student results obtained over a period of three years using this procedure.

TABLE 2
Student Spectrographic Results on Aluminum Samples

Sample No.	% *Si*	*Fe*	*Mn*	*Mg*
SA-360 Alcoa[a]	0.17	0.17	0.62	1.54
Av. of 6 student results	0.20	0.19	0.81	1.53
Av. dev. of the mean	0.02	0.01	0.02	0.01
SA-361 Alcoa	0.14	0.18	0.65	1.54
Av. of 6 student results	0.14	0.21	0.79	1.52
Av. dev. of the mean	0.02	0.01	0.02	0.01
SA-484 Alcoa	0.79	0.48	0.73	...
Av. of 6 student results	0.87	0.50	0.76	...
Av. dev. of the mean	0.03	0.01	0.04	...
SA-485 Alcoa	0.96	0.75	0.92	...
Av. of 4 student results	0.99	0.82	1.02	...
Av. dev. of the mean	0.04	0.02	0.03	...

[a] Aluminum Company of America analysis.

Flame Photometry. Finally, use is made of a Perkin Elmer Flame Photometer to complete the study of the field of emission spectroscopy. Figure 4 illustrates the equipment used. The samples analyzed are magnesium alloys with a lithium content varying from 1 to 20 per cent. The procedure used is as follows: Approximately a quarter-gram sample is weighed and dissolved in 5 ml. of 50 per cent hydrochloric acid. The excess acid is neutralized with ammonia until the solution is weakly acidic. The resulting solution is diluted

Figure 4. A Student Making a Measurement with a Flame Photometer

to an appropriate volume dependent upon the suspected lithium content. A series of standards of similar magnesium content and known lithium content are prepared and measured concurrently with the unknown sample using the flame photometer. A calibration curve is obtained by plotting the lithium content in p. p. m. versus the dial reading (per cent of standard) obtained on the flame photometer. Using this calibration curve and the dial reading for the unknown sample, the per cent of lithium in the alloy is determined.

ELECTROMETRIC METHODS

Polarography. In this experiment the instrument used is a Sargent-Heyrovsky Recording Polarograph (see Figure 5), and the samples analyzed are magnesium alloys. The procedure used is a modification of a method developed by Gull (*12*). A measure of two elements is obtained from each polarogram. Table 3 shows typical student results obtained on a few of the samples analyzed over a period of three years.

TABLE 3
Polarographic Results on Magnesium Alloys

Sample No.	*No. of student Determinations*	*% cadmium*	*% zinc*	*% copper*
320	8	1.98	2.24	...
	Av. dev. of mean	0.05	0.15	...
380	10	7.26	2.50	...
	Av. dev. of mean	0.18	0.18	...
381	14	6.66	4.04	...
	Av. dev. of mean	0.18	0.09	...
382	11	8.30	4.97	...
	Av. dev. of mean	0.16	0.15	...
523	6	4.75	...	0.85
	Av. dev. of mean	0.05	...	0.05

Amperometric Titration. In this experiment an analysis is made of an unknown chloride sample (Thorn Smith) by titration with silver nitrate using the conventional amperometric assembly with a Fisher Elecdropode to adjust the applied voltage to 0.06 volt. A 0.025 *N* silver nitrate solution is standardized against pure sodium chloride amperometrically, and then titrated against the unknown sample, in a solution containing KNO_3 as an inert electrolyte, gelatin to coagulate the precipitate, and bromthymol blue as a maximum suppressor. The galvanometer readings on the Fisher Elecdropode are plotted versus the ml. of silver nitrate solution added. At the start of the titration there is little change in the current measured with each increment of silver nitrate added because the silver ions are continually removed by the reaction $Ag^+ + Cl^- \rightarrow AgCl$. Once all the chloride ions are removed, the reaction $Ag^+ + e \rightarrow Ag^0$ occurs at the dropping mercury electrode and the current measured rapidly increases. The plot yields two sets of points, each set falling in a straight line, and the end point clearly occurs at their intersection. Hence, the volume of silver nitrate required is obtained, and the percentage of chloride in the unknown sample calculated. Using this procedure the student results obtained have been found to be comparable in accuracy with those obtained by the Volhard method.

Conductometric Titration. For this experiment a Leeds and Northrup Wheatstone bridge is supplied with audio-frequency alternating current by a Central Scientific Company Oscillator. The balance point is obtained by adjusting the bridge to minimum deviation from a linear trace on the tube of a Dumont Cathode-Ray Oscillograph.

The following procedure is used: A 50-ml. sample of vanilla is diluted with water and titrated by measuring the resistance after each addition of a 2-ml increment of standard sodium hydroxide solution. The reciprocal of the resistance value obtained (*i. e.*, the conductance) is plotted versus the ml. of sodium hydroxide added. Initially there is little change in the conductance ob-

Figure 5. A View of the Laboratory for Electrometric Methods Showing a Student Obtaining a Polarogram

An amperometric titration assembly may be seen in the foreground. The conductometric apparatus appears in the background

tained and a straight line can be drawn through this group of points. Once the end point has been reached, the unreacted hydroxyl ions causes a large increase in the conductance of the solution, and another straight line can be drawn through this set of points. The end point is determined graphically from the intersection of the two straight lines obtained. The samples titrated are several varieties of ordinary household vanilla, which contain vanillin, 3-methoxy-4-hydroxybenzaldehyde.

The phenolic group of vanillin can be titrated with sodium hydroxide and the amount of vanillin determined by this method. Since the solutions are dark colored, an ordinary colored indicator method is useless whereas the conductometric method yields satisfactory results. The student learns at least two basic facts from this experiment: the first being that an instrumental method may work where a conventional one may fail, and secondly that the price of vanilla is no criterion of its vanillin content.

Measurement of pH. The main purpose of this experiment is to acquaint the student with various methods of determining pH. The student first prepares a series of buffers covering the range from 4.4 to 9.4 at intervals of one unit. Then he measures the pH of each of the buffers using a LaMotte Roulette, a Hellige Comparator, and finally a Beckman pH meter. He is then given an unknown solution to determine its pH using each of the three instruments.

Potentiometric Titration. Although this type of titration is usually carried out in experiments in a physical chemistry course, the method is again used to illustrate its advantages. A determination of chromium in steel is made by oxidation of the chromium to the dichromate with ammonium persulfate and silver nitrate, and subsequent titration with a standard solution of ferrous ammonium sulfate. A total of three samples are run on each of the three following instruments, a Beckman Model G pH meter, a Fisher Titrimeter, and a Garman-Droz Titrimeter. A platinum electrode is used as the indicator electrode with a calomel electrode as a reference electrode. The use of three distinct instruments again serves to aid the student in evaluating the assets and defects of each type. The final phase of this experiment involves the titration of an unknown carbonate-bicarbonate mixture using a Beckman pH meter fitted with glass and calomel electrodes.

ADVANCED COURSE

The graduate student who is majoring in analytical chemistry is required to take an additional semester of instruction in instrumental analysis. Other graduate students who have had the first course or its equivalent may elect this additional training.

The advanced course, which is given to only a few students, is given in an informal manner. Each student chooses one of the three fields studied in the first course for specialized and advanced training for a semester. He consults with the instructor one hour each week, and must spend a minimum of two three-hour periods in the laboratory. This course frequently leads the analytical student to a choice of a subject for his doctoral thesis.

CONCLUSIONS

The experience of the past four years in teaching these courses has resulted in certain definite convictions or conclusions which may be of interest particularly to those who are contemplating the inauguration of formal courses in instrumental analysis.

The courses should be primarily laboratory, not demonstration, courses. This means that the number of students must be limited by the number of pieces of equipment which are available. The limitation thus imposed may be severe, but it is necessary if the student is really to profit from the opportunity presented. The student who uses the equipment himself is interested in his work; he acquires techniques which will be useful to him in his future work; he recognizes the limitations as well as the advantages of a particular method or instrument; and he may be able to avoid disappointment in his future research since his choice of that research will be based to some extent on his own experience.

It is hoped that the student will acquire the idea that measurement of any property of a substance is a possible analytical tool, and that a knowledge of the possible techniques will lead to the use of the best, and most economical, method of analysis.

The courses are liked by the students. This is particularly pleasing to the teacher of analytical chemistry who is usually accustomed to having graduates in chemistry leave the institution with an openly avowed distaste for analytical chemistry caused probably by a faint recollection of the sudden exposure in the sophomore year to the strict discipline of the subject.

Finally, a course in instrumental analysis may be begun with but a few pieces of equipment. It has been the experience of the authors that, once begun, the course makes a place for itself, and more equipment somehow becomes available. Indeed the problem before us now, at least in the first course, is how to choose which of the available methods and instruments should be used.

ACKNOWLEDGMENT

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TEACHING AMINO ACID FORMULATION

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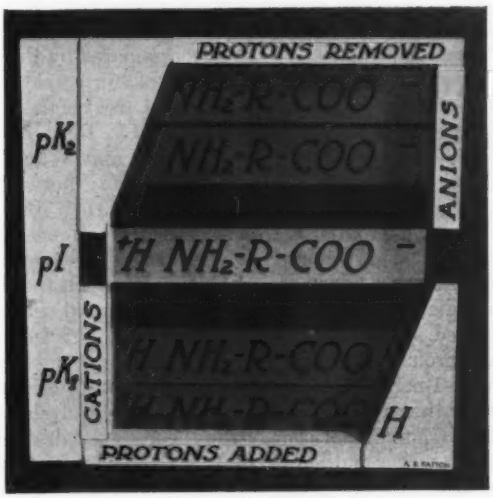
A CONCEPT of dipolar ions is necessary to an understanding of the behavior of amino acids and proteins. The author has designed a gadget which aids in explaining this concept to biochemistry students. It consists of a horizontal bar representing the amino acid or protein molecule, held in mortised grooves in a wooden frame with friction from a bullet catch in one end of the bar, which will slide from bottom to top, representing a shift in pH from 0 to 14, respectively. In the figure, the bar appears five times, because it is a multiple exposure showing various positions in which the bar is placed for class demonstration.

The theory involved is discussed in standard texts.[1, 2] An acid functions as a proton donor, and a base as a proton acceptor. An amino acid (or protein), represented by the formula NH_2—R—COOH, is potentially both an acid and a base, since the carboxyl groups (COOH) are proton donors and the amino groups (NH_2) are proton acceptors. As a consequence, amino acids exist as dipolar molecules, $^+NH_3$—R—COO^-, containing positively charged ammonium groups ($^+NH_3$), and negatively charged carboxylate groups (COO^-). When these opposite charges are in perfect balance, the amino acid exists as electrically neutral ions known as *zwitterions*. The pH at which this occurs is called the isoelectric point (pI). In salt-free solution it is called the isoionic point. At this pH the amino acid exists in its maximal state of dissociation.

In this zwitterion state, the $^+NH_3$ groups are the only potential proton donors (acid groups), and the COO^- groups are the only possible proton acceptors (basic groups). Hence, if protons are added to the system (titrating with HCl), the only possible site of acceptance is the COO^- groups, which are progressively converted to COOH. This is demonstrated by lowering the amino acid bar (hence the pH), while the diagonal on the frame shears off the ($^-$) sign and replaces it with (H). Since this results in a decrease in negative charges, the amino acid becomes progressively more cationic as the pH falls further below the pI.

When a half-molar equivalent of HCl has been added, the quantities [$^+NH_3$—R—COO^-] and [$^+NH_3$—R—COOH] are equal, and the dissociation constant $K_1 = [H^+]$. For the purposes of this demonstration activity coefficients are ignored. The corresponding pH is generally known as the pK_1. Progressing further, as the pH approaches 0, the formula of the amino acid approaches a limit of $^+NH_3$—R—COOH.



Conversely, if protons are removed from the system (titrating with NaOH), they are first stripped from the COOH groups until this supply is exhausted. The system is now back at the pI, and the amino acid exists in the form of zwitterions. With continued rise in pH (raising the amino acid bar), the only remaining proton donor is the $^+NH_3$ groups, which now begin to lose protons. This loss of protons decreases the positive charges, which are then overbalanced by the negative charges, with the result that the amino acid becomes progressively more anionic with increasing pH above the pI. When the half-way point is reached, [$^+NH_3$—R—COO^-] = [NH_2—R—COO^-], and $K_2 = [H^+]$. The corresponding pH is known as the pK_2. Beyond the pK_2, as the pH of the system approaches 14, the formula of the amino acid approaches a limit of NH_2—R—COO^-.

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THE PREPARATION OF 2-BROMONAPHTHALENE

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SATURATED and unsaturated 2-naphthalene acids have been prepared in this laboratory from 2-bromonaphthalene and tested for plant-growth regulating activity. Because 2-bromonaphthalene is so widely used for introducing specific groups into the naphthalene nucleus, an improvement in the method of preparation of this compound is of considerable interest. The method described here is suitable for the preparation of small or large quantities of 2-bromonaphthalene without the use of any special apparatus. A chromatographic procedure has been used to obtain a product which is sufficiently pure for use in Grignard reactions.

2-Bromonaphthalene is ordinarily prepared by the diazotization of 2-naphthylamine with sulfuric acid and sodium nitrite, followed by the usual treatment with cuprous bromide and hydrobromic acid.[1] In an alternative method, proposed by Newman and Wise,[2] 2-naphthylamine is first diazotized with hydrochloric acid and sodium nitrite. A stable complex is then formed with mercuric bromide and this complex decomposed by heating to 90°C. Wahl and Basilios[3] have recently reported a more satisfactory method, in which the dye intermediate 2-naphthylamine-1-sulfonic acid is first diazotized. The stable diazonium compound is treated with cuprous bromide and hydrobromic acid to obtain 2-bromo-1-naphthalenesulfonic acid. The sulfonic acid group is then removed by sulfuric acid hydrolysis and yields of 77 to 80 per cent of the theoretical value were reported. The method of Wahl and Basilios has been investigated in this laboratory in some detail and was found to give consistent yields of about 65 per cent when a technical grade of 2-naphthylamine-1-sulfonic acid was used as starting material.

Four hundred and forty-six grams (2 mols) of 2-naphthylamine-1-sulfonic acid (technical grade) is dissolved, with stirring, in 3600 ml. of aqueous sodium hydroxide (2.05 mols) contained in a 2-gallon battery jar or earthenware crock fitted with a mechanical stirrer. An aqueous solution of 138 grams (2 mols) of sodium nitrite is then added, with stirring, and the resulting solution is filtered.

One liter of concentrated (37 to 38.5 per cent) hydrochloric acid and 400 g. of crushed ice are placed in an insulated 5-gallon battery jar or earthenware crock, fitted with a mechanical stirrer, preferably of the wooden paddle-blade type. The filtered solution of sodium nitrite and sodium salt of 2-naphthylamine-1-sulfonic acid is then siphoned in, with stirring, and the temperature is maintained at 0–5°C. by adding crushed ice. The reddish-brown precipitate which forms is collected in a large Büchner funnel and washed with about 2 liters of iced water.

While the diazotization is in progress, a suspension of cuprous bromide in hydrobromic acid is prepared.[4] The damp cake of the diazonium compound is added portion-wise and stirred into 1100 ml. of the cuprous bromide (4.8 mols) suspension contained in a 5-gallon jar provided with an efficient mechanical stirrer. After the vigorous evolution of nitrogen has subsided, the mixture is heated to 95–100°C. on a steam bath and filtered hot through a large Büchner funnel. Some cuprous bromide will appear in the filtrate but this does not interfere with the next step in the preparation. The filtrate is poured back into the 5-gallon jar and 450 g. of potassium chloride is added with stirring. The resulting paste is allowed to cool to room temperature, then filtered with suction and washed with 1 liter of 20 per cent aqueous potassium chloride. The reddish-brown precipitate of 2-bromo-1-naphthalenesulfonic acid is air-dried overnight and then transferred to a 5-liter round-bottomed flask. A solution of 800 ml. of concentrated sulfuric acid (98 per cent) and 800 g. of crushed ice is prepared and added to the flask, with occasional shaking. A reflux condenser is attached to the flask, which is then placed in a heating mantle. The mixture is gently refluxed for 12 to 16 hours, then cooled to room temperature and poured onto about 2 kg. of crushed ice contained in a 2-gallon jar. The mixture is transferred to a 5-liter separatory funnel, and 2 liters of benzene are used to rinse the solid into the funnel. The mixture is shaken and the benzene layer is separated, then washed with water until the washings are neutral to litmus paper. After the benzene has been removed the product is distilled under reduced pressure. It boils at 100–101°C./2 mm. and solidifies to a slightly yellow solid which melts at 56.5–57.3°C.

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[4] The cuprous bromide is made from 1200 g. (4.8 mols) of copper sulfate ("Organic Syntheses," **1944**, Vol. 24, p. 23) using double the quantities, and is suspended in a solution of 300 ml. of 48 per cent hydrobromic acid and 800 ml. of water.

The average yield was 239 to 273 g. (58 to 66 per cent).

The product has a slightly yellow color which is not removed by redistillation or recrystallization and which becomes brown on exposure to air or light.[5] However, a pure white product can be obtained by dissolving 200 g. of the compound in 600 ml. of normal hexane and passing this solution through an activated alumina column (80–200 mesh) ½ in. in diameter and 3 to 4 in. in height. The column is then washed with an additional 600 ml. of normal hexane. The 1200 ml. of solution is evaporated to dryness and yields a white product melting at 56.8–57°C. (98 per cent recovery) which is sufficiently pure for use in Grignard reactions.

[5] This product apparently contains some amine compound. It cannot be used to form a Grignard reagent except in very low yields.

AN ATOMIC STRUCTURE MODEL

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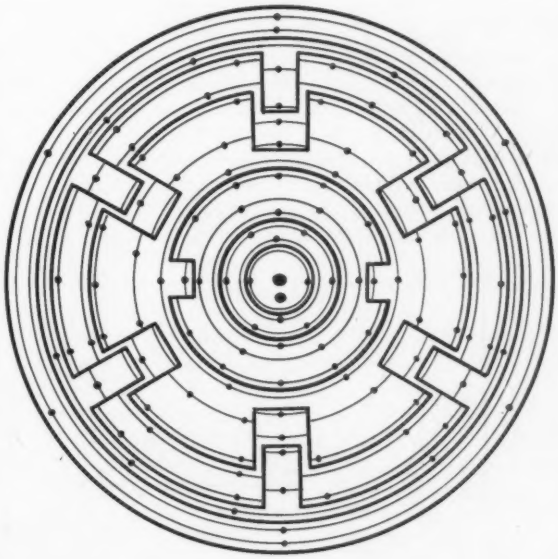
THE accompanying illustration is a drawing of a model which has been constructed by the author in an attempt to show the manner in which the electronic configurations of the various elements may be produced by adding electrons to a hydrogen atom.

In the actual model, the seven main electron shells are represented by discs of quarter-inch Upson Board each having two holes near the center (the larger black dots on the drawing) for mounting on two pegs driven into a wooden support. Two mounting holes are used so that the alignment of the discs will always be the same when they are assembled as shown in the drawing. The heavy black lines in the drawing represent the outlines of these discs some of which have cut-out portions for a reason to be explained later.

On these discs are drawn circles (the lighter circles in the drawing) representing the various subshells. These subshell circles are drawn with approximately uniform spaces between them on the discs in the following order: *1s, 2s, 2p, 3s, 3p, 4s, 3d, 4p, 5s, 4d, 5p, 6s, 4f, 5d, 6p, 5f, 6d, 7s,* and *7p*. It should be noted that the order of subshells *4f* and *5d* as well as that of *5f* and *6d* may be reversed in the cases of certain atoms and therefore these cannot be represented by means of this model. On account of the overlapping of the main shells, it has been necessary to cut out sections of some of the discs so that at least portions of all of the subshell circles will be visible when the model is completely assembled as shown in the drawing.

The discs should be painted in various pastel colors so that they may be distinguishable at a distance and the subshell circles drawn with black paint or crayon.

The heavy black dots on the subshell circles indicate possible positions of electrons. The positions of these dots have been arbitrarily fixed so that they will all be visible in the completely assembled model. In the model, these positions are indicated by wooden pegs



driven into the board. In using the model, electrons are represented by large wooden beads which are impaled upon the appropriate pegs depending upon which atom is to be represented. The pegs should be painted in a contrasting color so as to be visible, as should also the beads.

By means of this model the electronic configurations of the various elements can be shown, the principle that an added electron having a number of possible positions available to it will always occupy the one in which it will have the least possible energy (the one nearest the center) can be illustrated, and the difference between the transition elements and the others can be made clear.

SIMPLE MOLECULAR SPECTRA EXPERIMENTS

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WHILE there are many descriptions of laboratory exercises in atomic spectra suitable for physical chemistry classes,[1] the choice is more restricted in molecular spectra; especially is this so if simplicity in equipment and interpretation are prerequisites. The two following items, involving only the use of a visual spectrometer giving wave-length readings to 1 Å. or better, have been found very suitable in this laboratory. They illustrate many of the characteristic features in an absorption and an emission spectrum and they can be adequately described for present purposes in the form of an abbreviated instruction sheet. The emphasis throughout is on the determination of molecular factors of physicochemical interest.

THEORY

Simple molecules—and more particularly diatomic molecules such as I_2, which are being considered here—absorb visible or ultraviolet radiation because quanta of that order excite their electronic structures, *i. e.*, an electron is raised to an energy level higher than that found in the ordinary molecule. In both the normal and excited states the potential energy curves can be drawn representing the conditions in each state for various internuclear distances (r). These curves are of the form shown in Figure 1. In these curves the different vibrational energies which the molecule can acquire are shown by the levels at $n = 0$, $n = 1$, etc., where n is the vibrational quantum number. Most of the iodine molecules in the vapor at temperatures below 100°C. will be at the lowest vibrational level ($n = 0$) of the most stable electronic state.[2] It is from this molecular condition having least energy that they will start in the absorption process. If the atoms were bound by a force obeying Hooke's law, *i. e.*, by a perfectly elastic bond, their vibrations would be simple harmonic motions, the potential energy curve would be a parabola, and the successive vibrational energy levels would all be equally spaced. This is not found in practice as the restoring force decreases (slightly at first) on stretching the bond: consequently, the vibrational levels get closer together and with increasing values of r, $U(r)$ reaches an upper limit corresponding to the completely separated atoms. Clearly D_0 is the energy required to dissociate the molecule from its normal ground state, and this is one of the most important factors to be determined from the spectra. In absorption the molecule is excited to the n' levels of the upper electronic state, the different values of n' giving the separate bands. These energy changes are shown by the vertical lines from $n = 0$ in Figure 1. The vibrational absorption bands get closer together in going toward the violet in the spectrum owing to the convergence of the n' levels to a maximum energy value. Beyond the corresponding energy quantum L, there is a continuous (*i. e.*, nonbanded) absorption arising from the molecules dissociating into atoms with varying energies (*i. e.*, velocities) of separation.

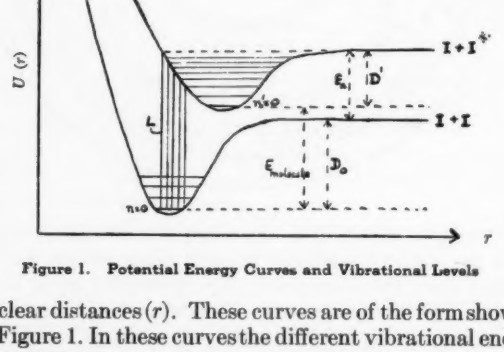

Figure 1. Potential Energy Curves and Vibrational Levels

In an emission spectrum, such as that of the C_2 molecule below, transitions occur from the various excited vibrational levels n' to the different values of n for the stable electronic state. This gives rise to a more involved pattern of vibrational bands. Under high resolution the individual bands would be seen to be composed of many fine lines, corresponding to the different rotational energy changes accompanying a single vibrational change. The present experiments are not concerned with this fine structure.

THE ABSORPTION SPECTRUM OF IODINE

Experimental. A Point-o-lite lamp is set at 60 cm. or more from the slit of the spectrometer, whose collimator, eyepiece, and cross wires are assumed in proper

[1] *E. g.*, H. W. THOMPSON, "A Course in Chemical Spectroscopy," Oxford University Press, London, **1938**. For brief accounts of this field, in order of difficulty, the student is referred to: (*a*) O. K. RICE, "Electronic Structure and Chemical Bonding," McGraw-Hill Book Co., New York, **1940**, Chap. IX. (*b*) S. GLASSTONE, "Textbook of Physical Chemistry," D. Van Nostrand Co., New York, **1940**, Chap. VIII. (*c*) A. E. RUARK AND H. C. UREY, "Atoms, Molecules and Quanta," McGraw-Hill Book Co., New York, **1930**, Chap. XII.

[2] The population in the other vibrational levels becomes appreciable as the temperature is raised and gives rise to further readily observed band sequences involving $n = 1, 2$, etc.

adjustment. An adequate opaque screen is conveniently arranged to reduce the illumination on the observer at the eyepiece. An iodine absorption vessel is interposed, at the correct height, between the Point-o-lite and the slit: this can be a clean air-filled spherical bulb, or cylindrical cell with plane ends, giving an absorption path of 12 to 20 cm. Preferably, the cylindrical cell is wound with a small (20-watt) heating coil, but otherwise the small amount of iodine vapor needed in the cell can be assured from the crystal or two placed in it by arranging the Point-o-lite rheostat at a suitable distance or by similarly adjusting a small Bunsen flame. The slits should be as narrow as is consistent with adequate background intensity, and the amount of iodine vapor controlled to give the clearest definition of the bands being measured. A condensing lens can greatly increase the illumination at the slit. Satisfactory conditions are very readily found in practice.

Careful measurements are made of the sharp band heads between 5432 Å. and 5100 Å.: the one near the former value corresponds to $n' = 27$, and the others must be numbered accordingly, *i. e.*, $n' = 28, 29, \ldots 50$, say.[3] After converting from wave lengths to wave numbers (*i. e.*, ω in cm.$^{-1} = 10^8/\lambda$ in Å.), and representing the wave-number difference between successive band heads by $\Delta\omega$, the values of n', λ (in Å.), ω (in cm.$^{-1}$), and $\Delta\omega$ may be tabulated.

Evaluation of the Data. (*a*) *The convergence limit.* The energy (in cm.$^{-1}$) corresponding to L might be estimated in a number of ways. Thus the wave length (λ_L) where the bands are replaced by continuous absorption might be directly observed, but this is not readily done satisfactorily. Again, a plot of ω against n' shows the gradual approach to the limit ω_L. Much more satisfactory, however, is a plot of $\Delta\omega$ for $(n' + 1) - (n')$ for the different values of n', against $(n' + 1/2)$. The points so obtained will show appreciable scatter about a straight line. Although they can be smoothed in various permissible ways, or the "best line" defined precisely by the method of least squares, it will suffice to draw the best straight line through them and extrapolate it to $\Delta\omega = 0$.[4] The total area under this curve from some chosen value of $(n' + 1/2)$ to $\Delta\omega = 0$ represents the wave-number difference between the mean for the bands n' and $(n' + 1)$, and the required limit. The area must, of course, be expressed in the wave number units appropriate to the plot. Assuming that the line accurately represents the trend in the $\Delta\omega$ values, the student should satisfy himself that this procedure is correct. Thus both ω_L and n'_L are found.

(*b*) *The vibrational frequency and the anharmonicity constant.* The vibrational levels in a molecule—and here we are referring to the electronically excited I_2 molecule—have the following energy values in wave numbers:

$$E_{n'} = G + \omega'_e\,(n' + 1/2) - \omega'_e\,x'_e\,(n' + 1/2)^2$$

ω'_e is the vibrational wave number (*i. e.*, the vibration frequency in units of cm.$^{-1}$) of the I_2 molecule for small vibrations in the excited state, and $\omega'_e \times 3 \times 10^{10}$ the corresponding vibration frequency in sec.$^{-1}$; x'_e is a small numerical factor, the anharmonicity constant, which measures the departures of the restoring force during the vibrations from Hooke's law. G is a constant referring the values to any appropriate point taken as the zero of the energy scale.

As the absorption starts from the normal I_2 molecule with fixed energy value ($n = 0$: Figure 1) the measured wave numbers should follow the relation:

$$\omega_{obs.} = \omega_{00} + \omega'_e\,(n' + 1/2) - \omega'_e\,x'_e\,(n' + 1/2)^2 \qquad (1)$$

ω_{00} is the wave number constant corresponding to the transition $n = 0$ to $n' = 0$. The student should show in his notes that from this equation the slope of the $\Delta\omega - (n' + 1/2)$ plot is given by $2\,\omega'_e x'_e$. Thus, from the line in (*a*) the value of $\omega'_e x'_e$ is obtained in cm.$^{-1}$ Using this value and substituting two reliable values of $\omega_{obs.}$ for n' values about 10 apart in equation (1), both ω_{00} and ω'_e are calculated. From the latter and $\omega'_e x'_e$, x'_e is obtained. These constants define the vibrational energy levels in the excited state and a check on ω_L is possible by calculating it from equation (1) with the value of n'_L found in (*a*).

(*c*) *Dissociation energies and force constants.* Clearly $(\omega_L - \omega_{00})$ is the dissociation energy D' for the excited molecules. Using the relation 1 cm.$^{-1}$ per molecule = 2.858 cal. per g. mol., conversion to the thermal units is made. A significant estimate of the same important factor is possible on the basis of the relation deduced from the energy levels given by equation (1) above; namely, $D' = \omega'_e/4x'_e$. This is compared with the previous value. Also, for the S. H. vibrator, $\omega'_e \times 3 \times 10^{10} = \frac{1}{2\pi}\,(f'/\mu)^{1/2}$, where f' is the elastic force-constant for the I—I bond in the excited state. If the masses in a diatomic molecule are A and B grams, μ = reduce mass = $AB/(A + B)$. This gives f' in dynes/cm.

Referring to Figure 1, it is seen that at the convergence limit, observed as ω_L, the iodine molecule dissociates into one normal and one excited atom:

$$I_2 \rightarrow I(^2P_{3/2}) + I^*(^2P_{1/2})$$

D_0 is the dissociation energy of the I_2 molecule in its normal electronic state and is a factor of immediate significance to the chemist. It is found from the energy relation $\omega_L = D_0 + E_a$, where E_a, is the excess energy of excitation of the I ($^2P_{1/2}$) atom. Its value is E_a = 7598 cm.$^{-1}$ and again D_0 is converted to cal./g. mol. It should be noticed that ω_e, the vibrational wave number in the ground state, is not observed in this spectrum. The Raman spectrum of I_2 gives it as 214.4 cm.$^{-1}$; this value is used to calculate f, the force con-

[3] This numbering is arrived at from an accurate study of the band sequences (*e. g.*, ref. in footnote 5) and its correctness is accepted in this experiment unless a much more detailed examination is made.

[4] The precise form of this convergence has been examined by W. G. Brown, *Phys. Rev.*, **38**, 709 (1931).

stant of the normal I_2 molecule. A summary of the results should include

$\omega_{00} = 15{,}590$ cm.$^{-1}$ $\omega_L = 20{,}040$ cm.$^{-1}$ $n'_L = (70)$

$\omega'_e x'_e = 0..\ 97$ cm.$^{-1}$ $\omega'_e = 131$ cm.$^{-1}$

$D' = 12.7$ kg.- cal./g. mol. $f' = 0.65 \times 10^5$ dyne/cm.

$D_0 = 35.5_8$ kg.-cal./g. mol. $f = 1.72 \times 10^5$ dyne/cm.

The significant indications provided by the last four factors and a comparison with those in other molecules (see, for instance, below) should be discussed by the student. Acceptable values[5] are quoted above.

THE SWAN BANDS AND THE C_2 MOLECULE

Hydrocarbons burning with a generous supply of oxygen give a characteristic emission spectrum, known after its discoverer as the Swan band system. This emission is responsible for the blue-green color of the cone of the Bunsen burner, and it is well-established that it arises from the C_2 molecule.

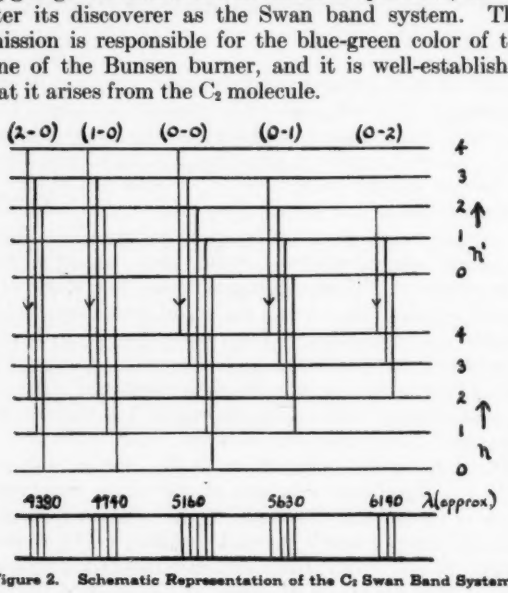

Figure 2. Schematic Representation of the C_2 Swan Band System

Experimental. A Bunsen burner is supported at least 40 cm. from the spectrometer; its height and position and that of a large condensing lens are adjusted so that an image of the green inner cone is focused on the entrance slit. This luminous cone will be brightest with about three-fourths the maximum air supply; more air will turn it blue and greatly reduce the C_2 band intensities. While the slit widths can be set say at 0.3 mm. during the preliminary observations, they should be reduced to give the sharpest definition of the features when measurements are being made. It is often helpful to be able to illuminate the cross wires in the eyepiece when wave-length settings on the weakest features are being made.

In this C_2 emission spectrum the electronically excited molecules are in different vibrational levels (n') and fall to different vibrational levels (n) in the ground state. Thus a large number of bands appear (each having rotational fine structure not considered here) and these group themselves in the spectrum in sequences, each sequence having a given value of $\Delta n = (n' - n)$. Diagrammatically the form of the spectrum may be represented as in Figure 2.

At least four sequences should be observed as broad bands against a darker background—and, most likely, the Na-doublet at 5893 Å. Each of the sequences is seen on careful examination to have a number of bright edges; these are the features to be measured, as they are the individual band heads. The student can usefully reproduce Figure 2, indicating on it the number and intensity of the observed band heads. Three or four wave-length readings of each head should be made and the mean values taken for conversion to cm.$^{-1}$

Evaluation of the Data. All the wave numbers are set into a table arranged:

	n 0	1	2	3	4
n'					
0					
1					
2					
3					
4					

It is noticed that the sequences form "diagonals" and that in any row the values fall in going from left to right; in a column, they increase on going from top to bottom. The student should account for these regularities in his notes.

The expressions for the vibrational energy levels in the excited (′) and ground states in wave numbers are:

$$E' = E_0' + \omega'_e(n' + 1/2) - \omega'_e x'_e(n' + 1/2)^2$$
$$E = E_0 + \omega_e (n + 1/2) - \omega_e x_e (n + 1/2)^2$$

where, again, the ω_e's represent the vibrational wave numbers for zero amplitudes, and the x_e's are the anharmonicity constants. For the transition $n' = 0 \rightarrow n = 0$

$$E_{00} = E'_0 - E_0 + 1/2(\omega'_e - \omega_e) - 1/4(\omega_e' x_e' - \omega_e x_e)$$

This energy change corresponds to a wave number E_{00} cm.$^{-1}$, the origin of the band system. The other band wave numbers are given by $(E' - E)$ for $n' \rightarrow n$; *i. e.*, $\omega_{n'n}$:

$$\omega_{n'n} = E_{00} + [n'\omega'_e - x'_e\omega'_e(n'^2 + n')] - [n\omega_e - x_e\omega_e(n^2 + n)]$$
$$= E_{00} + [n'\omega'_e - x'_e\omega'_e n'^2] - [n\omega_e - x_e\omega_e n^2] - [n'x'_e\omega_e - nx_e\omega_e]$$

As ω_e and ω'_e are of the same order, and both x_e and x'_e are $\sim 10^{-2}$, within the accuracy of these observations the last term in brackets can be neglected. Thus for constant n' the above expression reduces to:

$$\omega_{n'n} = \text{Constant} - [n\omega_e - x_e\omega_e n^2]$$

This relation must represent the wave numbers in any one row of the $n'n$ array. Substituting in it the values from any row in which three or more entries occur, both ω_e and x_e are calculated. The calculation can be

[5] MECKE, R., *Ann d. Physik*, 71, 103 (1923); and ref. in footnote 4.

repeated for any second row of three entries, and the mean values of these constants chosen. Using the relations previously quoted, the force constant f and the Morse value of the dissociation energy D for the ground state of the C_2 molecule are calculated. Similarly, with constant n,

$$\omega_{n'n} = \text{Constant}' + [n'\omega'_e - x'_e\omega'_e n'^2]$$

This relation clearly applies to any column in the array. It is used with any three entries in one column to calculate f' and D' for the excited state of the molecule. All the results, including the products $x_e\omega_e$, and $x'_e\omega'_e$, are summarized in a table with the appropriate units indicated in each case. A significant discussion can be based on them in the light of the accepted electronic configuration of the C_2 molecule ($^3\pi$) which represents a double bond. Precise data on this spectrum are readily available.[6] A consideration of the data obtained and evaluated in this simple way leads to one important suggestion. The force constant, f, for the ground state of the C_2 molecule is found to be 9.5×10^5 dyne/cm., in satisfactory agreement with the interpretation of the $^3\pi$ configuration as representing a double bond (compare, f = 9 to 10 units in simple ethylenic compounds). Herzberg[7] has argued in favor of a value for the dissociation energy of the ground state, D, equal to 83 kg.-cal./g. mol. (3.6 e. v.); Gaydon has tentatively used the same figure but recalls that the linear Birge-Sponer extrapolation leads to the much higher value of 160 kg.-cal./g. mol. The value obtained as above from the anharmonicity is 164 kg.-cal./g. mol. A figure of this order appears to conform far better to those in other diatomic molecules having double bonds than 83 kg.-cal./g. mol., which is more nearly characteristic of a normal single bond. Accordingly, in view of the value of the force constant and the well-known remarkable validity of the simple Morse potential function, the proposal is made that D really is in the region of 160 kg.-cal./g. mol. rather than half that value.

[6] *E. g.*, A. G. Gaydon, "The Dissociation Energies and Spectra of Diatomic Molecules," London, 1947; G. Herzberg, "Molecular Spectra and Molecular Structure," Prentice-Hall, Inc., New York, 1939, Vol. I.

[7] *Ibid.*, p. 472.

DEMONSTRATION OF OZONE FROM BOTTLED OXYGEN

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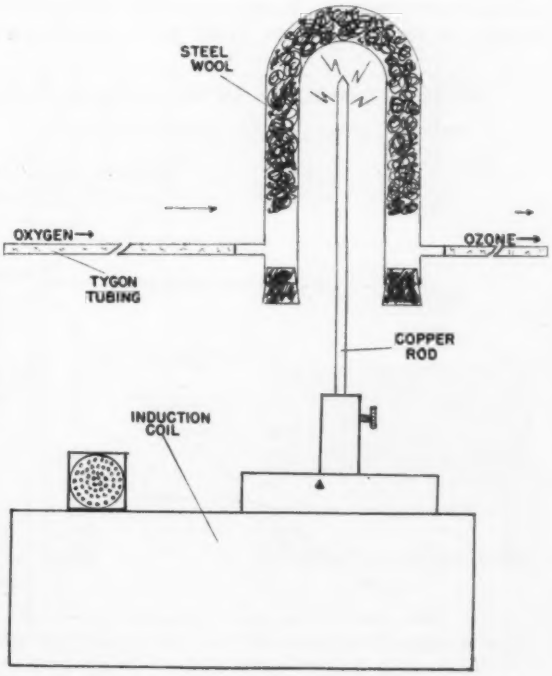
Ozone may be prepared by running a stream of oxygen through a piece of glass tubing about eight inches long containing a suitably sized reactangular file, or through a U-shaped gas drying tube packed with coarse steel wool, and toward either of which an induction coil is being discharged (see the figure). A silent-discharge ozonizer may be used for the same purpose.

Since rubber is quickly oxidized and destroyed by ozone it is important to use *plastic* tubing.

Ozone in the effluent gas can be detected by its odor as well as by the starch-KI reaction. Steel wool heated in a burner flame and held in a stream of oxygen (flowing through the generator with the induction coil switched off) will burn to a certain extent, but in a stream of ozone (now with the induction coil switched on) will burn about as rapidly as excelsior ignited with a match.



OSMOTIC PRESSURE AND MOLECULAR WEIGHT

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THE calculation of molecular weight from osmotic pressure is commonly illustrated, in textbooks of physical chemistry, by an application of the van't Hoff equation to a single determination. This equation may be written in the form

$$P = RTC$$

if P is the osmotic pressure in atmospheres, R is 0.0821, T the absolute temperature, and C the concentration in mols of solute per liter of solution. The equation implies that the calculation may be inaccurate unless it is known that the osmotic pressure actually is a linear function of the concentration. This equation was correctly regarded by van't Hoff as a limiting law, rigorously exact only for an ideal limiting state which solutions approach as they become more and more dilute. This implies that RT should be the limit approached by the ratio P/C as both C and P approach zero.

Perhaps the most practical use of the direct measurement of osmotic pressure has been to establish the molecular weights of proteins and synthetic polymers of large particle size. For solutions of such a material the osmotic pressure is rarely found to be a linear function of the concentration. In order to obtain the molecular weight it is necessary to make an extrapolation to infinite dilution before applying the limiting law. This procedure has been employed for some 20 years by those interested in proteins or other colloidal materials, but it is mentioned in very few textbooks. Because a simple extrapolation makes possible the correct use of a limiting law, the method might well be taught to students beginning the study of physical chemistry.

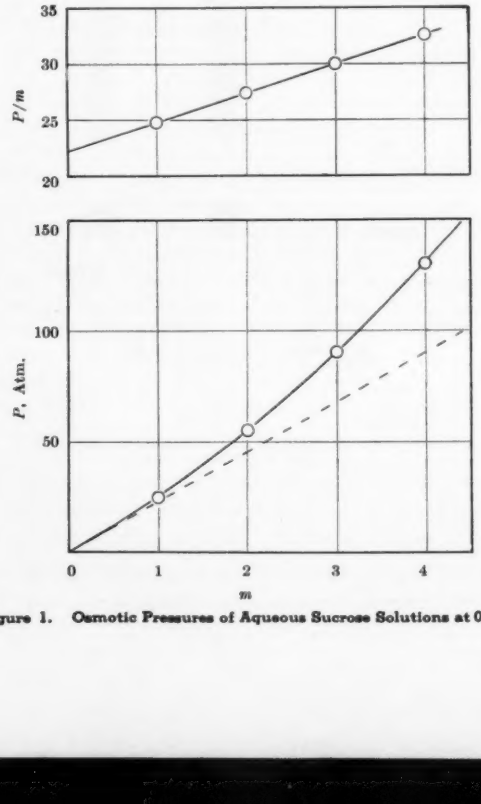

Figure 1. Osmotic Pressures of Aqueous Sucrose Solutions at 0°C.

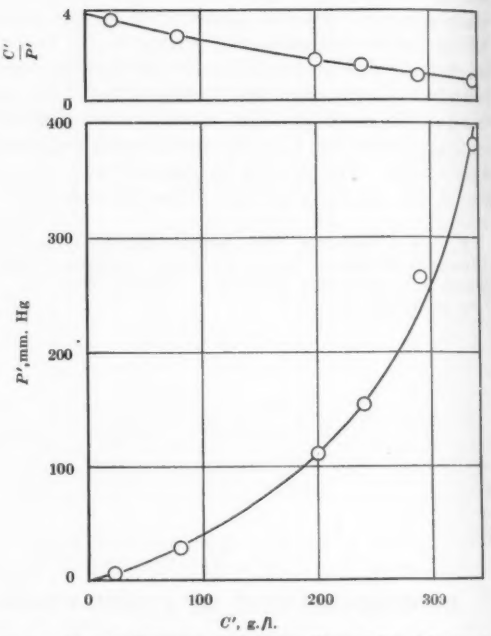

Figure 2. Osmotic Pressures of Aqueous Hemoglobin Solutions in a Saline Buffer at 0°C.

The fact that RT is the correct limiting slope of the relation between osmotic pressure and concentration can be justified empirically by a consideration of data to which the equation does not apply directly. Berkeley and Hartley (*1*) measured the osmotic pressures of concentrated sucrose solutions at 0°C., expressing the concentrations in grams of sucrose per 100 g. of water. On the basis of the accepted molecular weight of sucrose, their data have been tabulated (*2*) in terms of molality. In the lower part of Figure 1 the osmotic pressure (P) in atmospheres is plotted against the molality (m). The points fall on a curve which bends up as the molality is increased. At low molalities the curve approaches the broken line, which is a graph of Morse's modification of the van't Hoff equation,

$$P = RTm$$

In the upper part of Figure 1 the ratios, P/m, are plotted against m. These points lie on a straight line, and its intercept at zero molality is within 1 per cent of 22.4, which is the value of RT for 0°C. This shows that the Morse equation is a correct limiting law for these data, although none of the plotted points is fitted directly by that equation.

Similar plots, not reproduced here, were made from earlier data of Berkeley and Hartley (*3*), who then expressed the concentrations in grams of sucrose per liter of solution. After these were translated into concentrations (C) in mols per liter it was found that a plot of P/C against C was rather curved. A plot of C/P against C, while slightly curved, was straight enough to be extrapolated, and its intercept at zero concentration lay within 3 per cent of the reciprocal of 22.4. This shows that, within this margin of error, the van't Hoff equation is also valid as a limiting law for sucrose solutions at 0°C.

The use of a similar extrapolation for the calculation of a molecular weight may be illustrated by means of Adair's (*4*) measurements of the osmotic pressures of aqueous hemoglobin solutions[1] at 0°C. In the lower part of Figure 2 the osmotic pressures (P') in millimeters of mercury are plotted against the concentrations (C') in grams of hemoglobin per liter of solution. It is evident that a linear equation does not fit the data in the range shown. In the upper part of Figure 2 the ratio C'/P' is plotted against C'. The points are represented by a gently curved line which can be extrapolated to zero concentration; the intercept is 3.9. The van't Hoff equation, for this case, takes the form

$$P'/760 = 22.4\ C'/M$$

if M is the molecular weight. If we insert the limiting value of C'/P' and solve for M, we obtain

$$M = 22.4 \times 760 \times 3.9 = 66{,}000$$

A value not far from this figure for hemoglobin was first published by Adair in 1924, and later confirmed by Svedberg's measurements with the ultracentrifuge.

Extrapolations of the sort described here were applied to osmotic pressure data for other proteins by Adair and Robinson (*5*). Plots of P'/C' against P' were used by others (*6*).

[1] These solutions were equilibrated against a saline buffer mixture, and the membranes used were permeable to the ions of these salts as well as to water, but not to hemoglobin. Adair showed that a part of the observed pressure was due to the unequal distribution of ions. For the present purpose this ion pressure difference may be disregarded, since it vanishes in the extrapolation.

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(6) Scatchard, G., A. C. Batchelder, and A. Brown, *J. Clinical Investigation*, **23**, 458 (1944).

A RAPID METHOD FOR ROUTINE CHECKING OF MELTING POINTS

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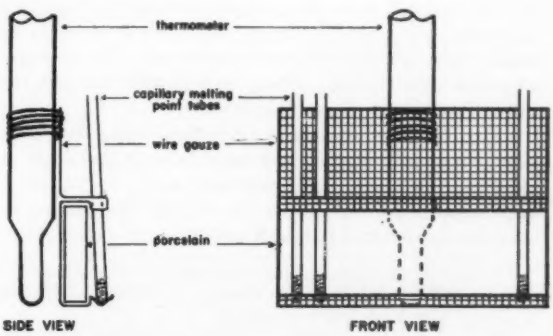
While working as a laboratory assistant in organic qualitative analysis, it was necessary to check the melting points of about two hundred derivatives prepared by students. The apparatus shown in the figure saved considerable time, as ten samples could be melted in one run. By arranging the samples in the order of increasing melting point and choosing samples with at least a ten-degree difference in melting points, it was possible to observe the range through which each individual sample melted.

A section of wire screen was bent to form a bracket to hold standard capillary melting point tubes and was wired to a thermometer. A small piece of white porcelain was provided as a background to facilitate visibility. The filled melting-point tubes were dropped into the holes in the screen with forceps and the thermometer assembly was lowered into the usual melting point bath, which was provided with a mechanical stirrer.



A SCHEME FOR THE QUALITATIVE IDENTIFICATION OF AMINO ACIDS[1]

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THE described scheme has been developed over a number of years and has been tested by numerous biochemistry students in the authors' classes. Seventeen of the naturally occurring amino acids distributed in mixtures containing from two to five components have been successfully identified. This scheme of analysis has been limited to the identification of pure amino acids in mixtures because the primary objective has been to acquaint the student with the more important properties and reactions of the amino acids.

Unknown mixtures are prepared to contain 400 mg. of each amino acid (200 mg. of cystine, proline, or tyrosine) as the free base, salt, or hydrate in the form of its optically active or racemic compound.

GROUP TESTS

I. Detection of Amino Acids by the Ninhydrin Reaction (9, 30). Dissolve 5 mg. of the amino acid mixture in 3 ml. of water, add 1 ml. of 0.1 per cent ninhydrin solution, and heat[3] the solution for 3 minutes. A blue to violet or red color denotes the presence of an amino acid. Acidify the solution with glacial acetic acid, add 4 ml. of chloroform, shake the mixture, and note the orange color of the chloroform layer.

II. Detection of Sulfur-Containing Amino Acids by Fusion with Sodium and Formation of Lead Sulfide.[4] *Cystine and Methionine.* Heat a clean 3-mm. cube of metallic sodium in a 3-inch soft-glass test tube until vapors form, add 25 mg. of the amino acid mixture in two portions, heat the tube to redness in a flame for 1 minute and allow the tube to cool to room temperature. Add 1 ml. of 50 per cent ethanol to destroy the excess sodium, heat the tube, and plunge the hot tube into 10 ml. of water in a beaker; filter the mixture containing shattered glass, acidify the filtrate with acetic acid, and add 1 drop of 2 *N* lead acetate solution. The formation of a black precipitate of lead sulfide indicates the presence of cystine and/or methionine.

III. Detection of Amino Acids Containing the Pyrrolidone Ring by Oxidation with Lead Dioxide and Red Color with p-Dimethylaminobenzaldehyde (17). Proline and Hydroxyproline. Dissolve 20 mg. of the amino acid mixture in 15 ml. of 0.3 *M* $Na_2HPO_4 \cdot 12H_2O$ buffer solution (pH 8.7), add 0.2 g. of PbO_2, and heat the mixture under reflux for 20 minutes. Filter the suspension and to 5 ml. of the filtrate add 5 ml. of *N* HCl and 1 ml. of 4 per cent *p*-dimethylaminobenzaldehyde in 95 per cent ethanol. Heat the solution. The immediate formation of a red color indicates proline and/or hydroxyproline.

IV. Detection of Aromatic Amino Acids by Nitration and Yellow-Red Color. Phenylalanine, Tryptophan, and Tyrosine (19). Add 1 ml. of concentrated HNO_3 and one drop of concentrated H_2SO_4 to 20 mg. of the amino acid mixture. Heat the mixture for 5 minutes. Cool the solution to room temperature and add 2 ml. of 18 *N* NaOH. The formation of a yellow color in the acidic solution and an orange to orange-red color in the basic solution indicates the presence of an aromatic amino acid. The color formed by phenylalanine may be faint in acidic solution but is readily detected in a basic one.

V. Solubility Separations. (*A*) *Absolute Ethanol.* Extract all of the available amino acid mixture twice with 10-ml. portions of absolute ethanol. Most of the proline and little of any other amino acid will dissolve. Evaporate the alcoholic extract and retain any solid for the proline confirmatory test. (*B*) *Water.*[5] Suspend the undissolved amino acid mixture (dried to remove ethanol) from (*A*) in 20 ml. of water, stir the suspension, and, if undissolved solid remains, heat the suspension with occasional stirring for 20 minutes at about 80° (water bath). Cool the mixture for 15 minutes in an ice-water bath and filter the suspension. Preserve the dried precipitate (cystine and tyrosine) and the filtrate (remaining amino acids).

VI. Precipitation of the Basic Amino Acids and Tryptophan with Phosphotungstic Acid (34, 35).[6] (*A*) *Arginine, Histidine, and Lysine.* To the aqueous solution obtained in Step V (*B*) add 37 ml. of 20 per cent phosphotungstic acid[7] and 1 ml. of 6 *N* HCl. Stir the

[1] A more detailed treatment of this scheme will be found in the authors' "Experiments in Biochemistry," McGraw-Hill Book Co. Inc., New York, 1951, (in press).

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[3] Unless otherwise stipulated a boiling water bath is used throughout to heat solutions.

[4] The Denis (*10*) fusion method is more hazardous and generally less satisfactory.

[5] Aspartic acid and leucine dissolve slowly but completely and in the presence of other amino acids, do not tend to reprecipitate during storage in the refrigerator. Tryptophan dissolves very slowly and, if undissolved, many interfere with the confirmatory test for tyrosine (Specific Test 5) by giving a brown color with Millon's reagent.

[6] If basic amino acids are absent as determined by test using 0.5 ml. of solution omit Step VI (*A*). If tryptophan is absent (Specific Test 6) omit Step VI (*B*).

[7] Phosphotungstic acid is purified by the method of Van Slyke (*35*). The volume of phosphotungstic acid reagent required to separate tryptophan from the basic amino acids by fractional precipitation is variable depending upon the number of the basic amino acids present. The precipitate may be colored brown or pink (if tryptophan is present) or change to a brown scum (if tryptophan alone is present). Glycine, alanine, and probably the other amino acid phosphotungstates do not precipitate within an hour.

mixture at intervals for 5 minutes, filter the suspension (if formed) with suction, and preserve the precipitate (A-1). Add 1 ml. of the phosphotungstic acid reagent to 5 ml. of the filtrate. If a precipitate forms combine this mixture with the remaining untreated filtrate and 22 ml. of the phosphotungstic acid reagent. Stir the mixture at intervals for 10 minutes and filter the suspension with suction. Test the precipitate (A-2) and the filtrate for tryptophan (Specific Test 6). Combine precipitates A-1 and A-2, if the latter contains little or no tryptophan, and preserve the mixture for the basic amino acids confirmatory tests (Tests 8, 9, and 10). If most of the tryptophan is in A-2 and relatiyely little in the filtrate preserve A-2 separately from A-1 for possible use if it becomes necessary to confirm the presence of lysine or glycine. (*B*) *Tryptophan.* To the solution from Step V (if basic amino acids are absent) or Step VI(*A*) add 0.5 ml. of 6 *N* HCl and 10 ml. of the phosphotungstic acid reagent. Cool the mixture for an hour in an ice-water bath with occasional stirring and filter the suspension. Clarify the filtrate (if turbid) by treating it with 0.5 g. of decolorizing carbon. Preserve the filtrate and the precipitate of tryptophan phosphotungstate for possible confirmation of lysine or glycine. (*C*) *Removal of Phosphotungstic Acid from Phosphotungstates* (*33*). Extract the filtrate from (*A*) or (*B*) with twice its volume of a 1:1 mixture of amyl alcohol and ether. Suspend each phosphotungstate precipitate in 10 ml. of 0.3 *N* HCl[8] and extract the suspension similarly with the amyl alcohol-ether mixture. If necessary, add with shaking a small aliquot of this solvent to promote separation of the aqueous (bottom) layer.

VII. Precipitation of the Acidic Amino Acids with Calcium Hydroxide-Ethanol (*14*). *Aspartic Acid and Glutamic Acid.* Heat to boiling the solution from Step V or VI (*C*), add an excess (1.0 or 1.2 g.) of carbonate-free calcium hydroxide, shake the mixture vigorously for a minute, and cool the suspension for 15 minutes in an ice-water bath. Add the mixture slowly with stirring to three volumes of 95 per cent ethanol,[9] shake it vigorously for 30 seconds, and filter the suspension (calcium salts of acidic amino acids). Acidify the filtrate to pH 4 with 0.5 *N* H_2SO_4, filter[9] the suspension of $CaSO_4$ and evaporate the filtrate to its original volume (preserve for Step VIII).

Suspend the precipitate (calcium salts of acidic amino acids) in water (volume half that of the original amino acid solution), heat the mixture to boiling over a free flame, filter the suspension (to remove excess calcium hydroxide and calcium carbonate), add the filtrate to three volumes of 95 per cent ethanol, cool the mixture for 15 minutes in an ice-water bath with occasional stirring, and filter the suspension. Preserve the precipitate for the aspartic acid and glutamic acid confirmatory tests (Tests 11 and 12).

VIII. Detection of Amino Acids Containing Adjacent Amino and Hydroxy Groups by Periodic Acid Oxidation (*31, 36*). *Serine and Threonine.* Add to the reaction tube of an aeration assembly 0.5 ml. of the amino acid solution (from Step VII), 3 ml. of 50 per cent K_2CO_3 and 2 drops of an antifoam agent (*e. g.*, Turkey red oil). Aerate (by means of suction) for 2 minutes to remove traces of ammonia, add 5 ml. of Nessler's reagent (*22*) to the receiver, add 2 ml. of 0.5 *M* periodic acid to the reaction tube and aerate. The appearance of a red color (test for ammonia) in the Nessler's reagent within 5 minutes' aeration indicates the presence of a hydroxy-amino acid in the unknown mixture.

IX. Detection of Ketone-Forming Amino Acids by Permanganate Oxidation (*6, 15*). *Isoleucine, Leucine, and Valine.* To 2 ml. of the amino acid solution (from Step VIII) add 1 ml. of 6 *N* H_2SO_4 and 1 ml. of 30 per cent $NaNO_2$ solution. Heat the mixture for 15 minutes to deaminize the amino acids and destroy the excess nitrous acid. Add NaOH to pH 4 (Congo red), 10 ml. of pH 6.8 buffer solution, 1 g. of solid $KMnO_4$, and a boiling stone. Distill (free flame) the mixture and collect the distillate in a receiver, containing 5 ml. of water and immersed in an ice-water bath, until all but about 3 ml. of the reaction mixture has distilled. To 3 ml. of the filtrate (preserve the remainder for Tests 15 and 16) add 3 ml. of water, 4 ml. of 95 per cent ethanol (acetone-free) and 4 ml. of concentrated H_2SO_4. Cool the mixture to room temperature, add 2 ml. of freshly prepared 6 per cent solution of salicylaldehyde in 95 per cent ethanol, and heat the mixture for 20 minutes at 55–60°. The appearance of a red color (test for acetone from valine or leucine or methylethylketone from isoleucine) indicates the presence of a ketone-forming amino acid in the unknown mixture.

SPECIFIC TESTS

Test 1. Proline (*16*). To 20 mg. of the solid isolated in Step V (*A*) add 5 ml. of glacial acetic acid and 1 mg. of isatin. Heat the mixture on a boiling water bath. The appearance of a blue color (within 5 minutes) confirms the presence of proline in the unknown mixture.[10]

Test 2. Hydroxyproline (*26*).[11] To 0.2 ml. of the amino acid solution (from Step VI) add 1 ml. of 0.01 *M* CuSO, 1 ml. of 3 *N* NaOH, and 1 ml. of 6 per cent H_2O_2. Allow the mixture to stand for 5 minutes with frequent shaking. Heat the mixture for 5 minutes, cool

[8] HCl is added to liberate the phosphotungstic acid from the phosphotungstates and to prevent emulsification during the extraction with amyl alcohol-ether.

[9] Upon standing a sticky semicrystalline precipitate may be formed which adheres to the walls of the flask and clogs the pores of the filter paper. A similar type precipitate may result if the ethanol is added to the lime suspension. Under these circumstances decant the solvent, redissolve the calcium salts of the acidic amino acids, and repeat the precipitation process as described.

It has been observed that no precipitation occurs under the described conditions if the proportion of ethanol to amino acid solution is increased from 3:1 to 9:1 (*5*). The calcium salt of aspartic acid precipitates more readily and more completely than that of glutamic acid.

[10] The test is specific for proline under the described conditions since hydroxyproline and other amino acids which give a pink color have been separated.

[11] The Morse (*28*) test is specific but, as reported by McFarlane and Guest (*26*), explosions occur.

the solution to room temperature, add N HCl dropwise until the deep blue color is discharged and add 0.3 ml. of N HCl. To 2 ml. of this solution add 2 ml. of freshly prepared 1 per cent isatin solution and 2 ml. of N HCl. Heat the mixture. The appearance of a red color (within 5 minutes) confirms the presence of hydroxyproline in the unknown mixture.

Test 3. Cystine (*32*). Dissolve 1 mg. of the precipitate obtained in Step V (*B*) in 5 ml. of 0.1 N NaOH, add 1 ml. of 5 per cent NaCN solution in N NaOH, and allow the mixture to stand for 10 minutes. Add 1 ml. of a 0.5 per cent solution of sodium 1,2-naphthoquinone-4-sulfonate (*12*) and 5 ml. of a freshly prepared 10 per cent Na_2SO_3 solution. Allow the mixture to stand for 10 minutes. Add 1 ml. of freshly prepared 2 per cent $Na_2S_2O_4$ solution. The appearance and persistence of a red color confirms the presence of cystine in the unknown mixture.

Test 4. Methionine (*25*). To 0.5 ml. of the amino acid solution (from Step VI) add 2 ml. of water, 3 ml. of 3 N NaOH and 1 ml. of a freshly prepared 1 per cent sodium nitroprusside solution. Heat the mixture for 5 minutes at 35–40° (water bath), cool the solution in an ice-water bath, and acidify the solution with 3 ml. of 6 N HCl added slowly with shaking. The appearance of a red color confirms the presence of methionine in the unknown mixture. Histidine and tryptophan, which react similarly, do not interfere since they were removed in previous steps.

Test 5. Tyrosine. To 1 mg. of the precipitate obtained in Step V (*B*) add 1 ml. of water and 2 drops of Millon's reagent. Heat the mixture. The appearance of a bright red color (within 1 minute) confirms the presence of tyrosine in the unknown mixture.

Test 6. Tryptophan.[12] To a drop of the amino acid solution (Step VI) add 2 ml. of water and 2 drops of the Hopkins-Cole reagent (*4*). Add concentrated H_2SO_4 in such a manner that it forms a layer below the aqueous solution. Heat the solution. The appearance of a violet to black ring at the interface of the layers (within 2 minutes) confirms the presence of tryptophan.

Test 7. Phenylalanine (*20*).[13] Evaporate 2 ml. of the amino acid solution (Step VI) to dryness and add 2 ml. of nitrating agent (10 g. of KNO_3 per 100 ml. of concentrated H_2SO_4). Heat the mixture for 20 minutes with occasional (cautious) stirring, cool the solution to room temperature, add 3 ml. of water, and cool the mixture for 5 minutes in an ice-water bath. Add 4 ml. of 20 per cent $NH_2OH \cdot HCl$, allow the mixture to stand for a minute, and add 10 ml. of concentrated NH_4OH in small portions. Allow the mixture to stand at room temperature. The appearance of a violet color (within 30 minutes) confirms the presence of phenylalanine in the unknown mixture.

Test 8. Arginine (*37*). To 0.1 ml. of the solution prepared in Step VI (*C*) add 5 ml. of water, 1 ml. of 6 N NaOH and 1 ml. of 0.02 per cent α-naphthol in 20 per cent ethanol. Cool the mixture for 5 minutes in an ice-water bath and add 2 drops of NaOBr (2 per cent bromine in 1.5 N NaOH). The immediate appearance of a red color confirms the presence of arginine in the unknown mixture.

Test 9. Histidine (*21*). To 0.1 ml. of the amino acid solution prepared in Step VI (*C*) add 2 ml. of water and bromine solution (1 per cent bromine in 33 per cent acetic acid) dropwise until a light yellow color persists. Allow the solution to stand for 5 minutes, add 2 ml. of ammonia solution (3.3 per cent ammonium carbonate in 10 N NH_4OH) and heat the mixture. The appearance of a blue-violet color (within 5 minutes) confirms the presence of histidine in the unknown mixture.

Test 10. Lysine (*7, 24*). (*A*) To 10 ml. of the amino acid solution prepared in Step VI (*C*) add 15 ml. of a saturated aqueous solution of picric acid. Shake the mixture and cool it in an ice-water bath or the refrigerator. The appearance (within 24 hours) of yellow needles (lysine picrate) indicates the presence of lysine in the unknown mixture. (*B*) To 0.5 ml. of the amino acid solution prepared in Step VI (*C*) or, if histidine is present, 20 mg. of the precipitate (lysine picrate) add 1 ml. of saturated bromine water and 2 drops of 6 N HCl. Allow the mixture to stand for 5 minutes, add 5 per cent $NaAsO_2$ solution until the color is discharged, add Na_2CO_3 to a basic test, heat the mixture for 2 minutes, add 0.5 ml. of a phosphotungstic acid-phosphomolybdic acid reagent (*13*) and heat the mixture. A green color which changes to deep blue or blue-green in presence of picric acid within 5 minutes confirms the presence of lysine in the unknown mixture. A green color which deepens to blue after 20 minutes' standing denotes arginine. In the presence of histidine an interfering purple color formed on addition of Na_2CO_3 interferes with the test for lysine.

Test 11. Glutamic Acid (*18*). Dissolve the precipitate prepared in Step VII in water, add 1 M oxalic acid with stirring to pH 3.5 (Congo red), filter the suspension of calcium oxalate, saturate the filtrate (cooled in an ice-water bath) with hydrogen chloride from a generator, and allow the solution to stand in an ice-water bath or in the refrigerator. The formation (within 24 hours) of needle-like crystals (glutamic acid hydrochloride) confirms the presence of glutamic acid in the unknown mixture.

Test 12. Aspartic Acid (*29, 33*).[14] (*A*) Heat about one-sixth of the solution from Test 11 for 5 minutes on a boiling water bath to remove excess HCl. Add 10 ml. of water, 1 ml. of concentrated H_2SO_4, and 1 ml. of saturated bromine water. Heat the mixture for 5 minutes, cool the solution to room temperature, and add 1 ml. of N KBr solution and 2.5 ml. of 1.5 N

[12] The Winkler (*38*) modification is more sensitive but need not be employed since any small amount of tryptophan undetected after the phosphotungstic acid treatment would not interfere with tests for other amino acids.

[13] The green color formed by treatment of phenylacetaldehyde (formed from phenylalanine) with ethanol and sulfuric acid (*22*) was not found to be a sufficiently sensitive test.

[14] The reaction of β-naphthol with malic acid (formed by deamination of aspartic acid) to give a yellow-colored substance with blue fluorescence (*11*) could not be employed because of inhibition of fluorescence by traces of nitrous acid.

$KMnO_4$ solution. Allow the solution to stand for 10 minutes and add 6 per cent H_2O_2 dropwise until the solution is decolorized. Attach an aeration assembly to the reaction tube, place the receiving tube in an ice-water bath, add 5 ml. of clear solution of 2,4-dinitrophenylhydrazine in 2.4 *N* HCl in the receiving tube, place the reaction tube in a boiling water bath, and aerate (by means of suction) until about 8 ml. of liquid has distilled. An orange-colored precipitate of glyoxal-2,4-dinitrophenylosazone indicates the presence of aspartic acid in the unknown mixture. Confirm the identity of the precipitate by washing it on a small filter, dissolving it by pouring a small volume of pyridine repeatedly over the precipitate and adding about 10 volumes of water and 2 ml. of 6 *N* NaOH to the filtrate. The solution should be blue colored.

(*B*) Evaporate the remainder of the solution from Test 11 to a sirup, add water, re-evaporate the solution, dissolve the sirup in 5 ml. of hot water, and add 0.5 g. of $CuCO_3$ (or 5 ml. of saturated cupric acetate solution) in small portions with stirring and continued heating. Filter the suspension and allow the filtrate to stand overnight in the refrigerator. The formation of fine blue needles (or pale blue precipitate) with a violet tinge indicates the formation of cupric aspartate hydrate and confirms the presence of aspartic acid in the unknown mixture.

Test 13. Threonine (*31*).[15] To 0.1 ml. of the solution prepared in Step VII add 2 drops of antifoam agent, 5 ml. of 0.5 *M* $NaHCO_3$, 7 ml. of 0.1 *N* sodium arsenite in 2 per cent $NaHCO_3$, and 1 ml. of 0.5 *M* periodic acid. Attach an aeration assembly to the reaction tube, place 10 ml. of concentrated H_2SO_4, and 10 mg. of *p*-hydroxydiphenyl in the receiving tube and aerate (by means of suction). The appearance of a violet color (within 20 minutes) indicates the formation of acetaldehyde and the presence of threonine in the unknown mixture. Preserve the solution in the reaction tube for Test 14.

Test 14. Serine (*8*). Acidify the solution (from Test 13) in the reaction tube with 6 *N* acetic acid, attach the aeration assembly with a clean receiving tube containing 10 ml. of water, place the receiving tube in an ice-water bath, place the reaction tube in a boiling water bath and aerate (by means of suction) until (about 20 minutes) the volume of the solution in the reaction tube has been reduced to about 5 ml. To 3 ml. of the solution in the receiving tube add 5 ml. of water, 3 ml. of concentrated H_2SO_4 and 10 mg. of chromotropic acid (1,8-dihydroxynaphthalene-3,6-disulfonic acid). Heat the tube. The appearance of a violet-rose color (within 20 minutes) confirms the presence of formaldehyde in the solution and serine in the unknown mixture.

Test 15. Leucine and Valine (*6*). To 1 ml. of the solution prepared in Step IX add 10 ml. of 6 *N* NaOH and 1 ml. of 6 per cent salicylaldehyde in ethanol. Heat the solution. The appearance of a red color (within 15 minutes) confirms the presence of acetone in the solution and leucine or valine in the unknown mixture.

Text 16. Isoleucine (*6*). To 5 ml. of the solution prepared in Step IX add 100 ml. of water, 5 ml. of concentrated H_2SO_4, and 35 ml. of Denigès' reagent (15 per cent $HgSO_4$ in 6 *N* H_2SO_4 (*9*)). Reflux (free flame) the mixture for 30 minutes and filter the suspension if a precipitate (acetone-mercuric sulfate complex) is formed. To 5 ml. of the filtrate add 3 ml. of 95 per cent ethanol, 2 ml. of concentrated H_2SO_4, and 1 ml. of 6 per cent salicylaldehyde in ethanol. Heat the solution. The appearance of a red color (within 15 minutes) confirms the presence of methylethylketone in the solution and isoleucine in the unknown mixture.

Test 17. Alanine (*2*). To 0.5 ml. of the solution prepared in Step VII add 3 ml. of water, 0.1 *N* NaOH to pH 6.5–7.0 (bromthymol blue) and 2 ml. of 1 per cent ninhydrin solution. Heat the mixture for 3 minutes. Attach an aeration assembly to the reaction tube, place 5 ml. of concentrated H_2SO_4 and 10 mg. of *p*-hydroxydiphenyl in the receiving tube and aerate by means of suction. The appearance of a pink to violet color (within 15 minutes) in the receiving tube solution confirms the presence of acetaldehyde in the solution and alanine in the unknown mixture.

Test 18. Glycine (*1*).[16] Acidify the solution in the reaction tube (from Test 17), aerate the solution and test the filtrate as described in Test 14. The appearance of a violet-rose color (within 20 minutes) confirms the presence of formaldehyde in the solution and glycine in the unknown mixture.

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[15] On treatment with NaOBr and KI_3, alanine and threonine give a positive but not always reliable iodoform test. Most of the tests (*27*) described for alanine and glycine have been investigated but none has been found as satisfactory as the ones given in Tests 17 and 18. Acetaldehyde, but not formaldehyde, is removed by aeration at pH 7.

[16] Formaldehyde may be detected by adding an equal volume of concentrated H_2SO_4 and chromotropic acid to the reaction tube but this procedure is less satisfactory than the one described. The *o*-phthaldialdehyde test (*39*) is sensitive and relatively specific but the reagent is difficult to prepare and is not available commercially. The precipitation of glycine as its trioxalatochromate (*5*) is specific but often leads to uncertainty owing to the tendency of the reagent to precipitate in the absence of glycine.

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GUNNAR HÄGG

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From the close of the phlogistic period, *i. e.*, from the incumbency of Tobern Bergman (1735–84),[1] Upsala has been a notable center of chemical training and research. Appointment to a professorship in this faculty is indeed a recognition of proved ability. Gunnar Hägg has occupied the chair of general and inorganic chemistry at this Swedish university since 1936.

Born at Stockholm on December 14, 1903, he entered the University of Stockholm in 1922. In 1926 he studied at University College, London, under F. G. Donnan[2] and then returned (1927) to Stockholm where he began to work in X-ray crystallography under A. Westgren. After the Ph.D. was conferred by the University of Stockholm in 1929, Dr. Hägg was appointed lecturer there in general and inorganic chemistry. He also became associated with the theoretical division of the Metallographic Institute (Stockholm), which was directed by Westgren.

The main emphasis in his earlier researches was on crystal structure by means of X-ray diffraction. He studied especially the metallic phases produced by transition elements and nonmetals such as hydrogen, boron, carbon, and nitrogen. In connection with these investigations he also took up the martensite problem and the nitriding of steel. Solid solutions of the inter stitial type, particularly the so-called subtraction solu tions (such as are found in pyrrhotite, *i. e.*, the soli solution of sulfur in FeS, the spinels leading to γ Al_2O_3 and γ- Fe_2O_3, the tungsten bronzes), also re ceived his attention. He made studies of layer lattice and the order-disorder phenomena found in them The structure of the dithionate ion represents anothe phase of his researches.

The work at Upsala is now being conducted alon different lines with the collaboration of students an others. The subjects include the determination of th structures of metal oxides, particularly oxides of moly denum and tungsten; structures of polymolybdate which have led to the interpretation of the anions of th para-, tetra-, and dimolybdates; metallic borides structures of borates and boric oxide; and developme of electrochemical methods, especially in polarography

He wrote "Kemisk Reaktionslära" (first edition 194 fourth edition 1948) to satisfy the need of a te book presenting the theoretical bases of analytic chemistry. A translation of this excellent text in German by Hans Baumann was issued in 1950 at Ba with the title "Die theoretischen Grundlagen d analytischen Chemie."[3]

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AN ANCIENT HINDU CONCEPT OF A CHEMICAL LABORATORY

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THE most ancient Indian medical treatises are those of Charak and Susruta. The name of Vāgbhaṭa is considered to be the third great name in ancient Indian medical science. The work "Rasaratnasamuccaya" is usually ascribed to Vāgbhaṭa though some scholars attribute it to Acvinikumāra or Nityanātha.[1] The probable date of this book, according to Dr. Jolly, is 1300 A.D.[2] This work is divided into 30 chapters. Chapters I to V deal with various metallic preparations used in Ayurvedic practice of medicine. In these chapters their occurrence, mode of preparation, chemical, physical, and physiological properties are described. Chapter VI deals with the qualities of the teacher and the taught and various kinds of prayers and offerings to be performed by them. In Chapter VII is given the concept of a laboratory. Chapter VIII deals with certain definitions. The description of chemical apparatus and instruments is given in Chapters IX and X. In Chapter XI certain chemical processes are described. The rest of the Chapters XII to XXX deal with various diseases and prescriptions for their cure. The rest of this paper is a translation of some of the verses in Chapter VII.

The laboratory should be built in a place which is free from disturbances of all kind. The surroundings should be beautiful and all varieties of drugs and chemicals should be available near-by. Near the laboratory should be situated a well or a water tank. (Verse 1)

The laboratory should be encircled by a compound wall. It should consist of eight divisions or eight portions. In the east should be established the deity—Shankara. In the southeast should be furnaces and other arrangements for making fire. The south should have many types of pestle and mortar and other devices for crushing and powdering solid material. The metal or mineral testing section and the workshop for the construction of new apparatus should be placed in the southwest. The west portion of the laboratory should be equipped with arrangements for washing. In the northwest should be arrangements for drying. The north portion of the laboratory should be reserved for the preparation of drugs and chemicals and the northeast should be used for storing the finished products. (Verses 2, 3, 4)

[1] KEITH, A. B., "A History of Sanskrit Literature," Oxford, p. 512.

[2] JOLLY, J., "Festschrift Windisch," p. 192.

The equipment of the laboratory should consist of:

1. All kinds of instruments and apparatus both on the bigger scale and in portable sizes.
2. Various types of distillation apparatus.
3. Apparatus for extraction of oils.
4. Several types of vessels made of metal and clay for carrying and storing water.
5. Two bellows.
6. Two long tubes of metal or two hollow bamboos.
7. Small vessels (crucibles) made of gold, iron, copper, *kansya* (an alloy), and stone.
8. Leather apparatus.
9. All other kinds of instruments and apparatus used for preparing and studying drugs.
10. Crushing instruments—stone mills, pestles, and mortars of all shapes and sizes made of stone and metals.
11. Sieves with thousands of holes.
12. Instruments for cutting metals.
13. Big and small vessels of iron and their stirring rods. Etc., etc. (Verses 5, 6, 7, 8, 9)

For sieving powders there are three types of sieves. The one made of bamboo sticks tied together with strong thread is used for sifting coarse powders. The second type of sieve is also made from the bamboo. The third type is four fingers (3 in.) high and one finger spread (9 in.) wide. Its sides are constructed of goatskin and in the middle is fixed either a cloth or horsehair. It is used for sieving fine powders. (Verses 10, 13)

Five kinds of clay, wheat husk, cotton, wild combustible material, three types of metals, urine of animals, fundamental vegetable drugs, cow dung, sand, white sand, coal, and coke should be collected in the laboratory. (Verses 14, 15, 16)

The laboratory should possess bottles and cups made up of glass, iron, clay, and shells. (Verse 18)

Small vessels made up of shell, small conch shells, small knives, instruments for cutting, vessels for heating, and brooms for cleaning should be collected. In short, all the things required or likely to be required for the study should be gathered and collected. (Verses 21, 22)

In this laboratory should be collected physicians who have a thorough knowledge of Nighanṭu (Medical dictionary), are well versed in the art of preparing medicines, and are conversant with all the languages. (Verse 24)

THE MOLECULAR WEIGHT OF A PROTEIN

A Student Determination of Osmotic Pressure

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ALTHOUGH the measurement of osmotic pressure is one of the standard methods for determining molecular weights of such macromolecular substances as proteins, the common physical chemistry laboratory texts describe no experiments to illustrate this method in elementary laboratory. The readily available descriptions of methods[1] are too elaborate and too tedious for student use, principally because membranes of general applicability are tedious to prepare, fragile, and of such low porosity that the osmotic equilibrium is approached very slowly.

A quantitative osmotic pressure determination would be an especially valuable illustration of colligative properties to a premedical physical chemistry class, particularly if applied to a system of biological interest. However, the limited laboratory time available to students in such courses makes it essential that the experiment be easily completed within one laboratory period. This can be accomplished with a macromolecular solute since the demands on the membrane are not too great.

With rather modest demands on equipment, time, and technique, this experiment gives for gelatin solutions molecular weights which, although not precise, agree in order of magnitude with each other and with reported values.

A "Hengar tube," *i. e.*, a test tube-shaped porous ceramic vessel which is used in a modified Kjeldahl digestion procedure[2] and is available from laboratory supply houses, is impermeable to gelatin and sufficiently porous for osmotic equilibrium through it to be reached in less than two hours. The pressure is simply determined by the equilibrium difference in height of the levels of the solution and the pure water. A cathetometer was available for this experiment and was used for the height measurement, but a simple ruler could be used, although with somewhat less ease and somewhat less (yet adequate) precision. With 6-mm. tubing for the solution to rise in, it can be shown that the change in concentration of the solution due to water passing through the membrane is negligible. During preliminary trials run to establish conditions for the experiment, changes in the water level outside the membrane were followed by corresponding adjustments in the level of the solution, demonstrating the reversibility of the osmotic process.

The Hengar tube loses its porosity if allowed to dry out after repeated use in this experiment; it may be restored by immersion in boiling water for an hour or in water at room temperature overnight, or by performing the experiment at about 50°. The temperature of the experiment must be controlled carefully; otherwise, fluctuations in the pressure of the air trapped in the apparatus vary the solution level, obscuring the slow pressure change due to the approach to osmotic equilibrium.

DIRECTIONS

Apparatus. Five-inch Hengar tube fitted with a 1-hole stopper and glass tube, 800-ml. tall-form beaker, 100-ml. volumetric flask, paraffin wax, gelatin, cathetometer.

Procedure. Weigh between 0.5 and 1.0 g. gelatin to the nearest 0.005 g., dissolve in hot water (distilled), and dilute to 100 ml. Fill the Hengar tube to the end of the narrow part with gelatin solution and insert the stopper bearing the glass tube, making sure that the tube dips at least 1 cm. into the solution. Seal over the rubber stopper contacts with paraffin and immerse the Hengar tube and stopper in distilled water, supporting it by a clamp on the glass tube. Maintain the entire system at a temperature of about 35°C. to prevent gel formation (the elevated temperature also hastens the reaching of equilibrium). Focus the telescope of the cathetometer on the meniscus in the glass tube above the Hengar tube. Sight on the meniscus from time to time until its position becomes constant. Since osmotic pressure depends upon temperature, there will be some variation of the level even after equilibrium is reached if the temperature is changing. Read on the cathetometer the height of the meniscus and the height of the water outside the tube, approaching both readings from the same direction to allow for play in the instrument. Read the temperature of the water in contact with the membrane.

Calculations. From the difference in levels calculate the osmotic pressure in dynes/cm.[2] and in atmospheres. Use 1.00 g./ml. as the density of the solution. From the temperature, osmotic pressure, and composition of the solution calculate the molecular

[1] DANIELS, F., J. H. MATHEWS, AND J. W. WILLIAMS, "Experimental Physical Chemistry," 4th ed., McGraw-Hill Book Co., Inc., New York, 1949, pp. 439–42. R. H. WAGNER, "Physical Methods of Organic Chemistry," edited by A. WEISSBERGER, Interscience Publishers, Inc., New York, 1945, Vol. I, pp. 265–75.

[2] SWIFT, E. H., "Introductory Quantitative Analysis," Prentice-Hall, Inc., New York, 1950, p. 287.

weight of gelatin. Consider the error due to some water passing through the membrane before equilibrium is reached.

STUDENTS' RESULTS

The table shows the gelatin concentration used and the difference in level found by six pairs of students together with the molecular weight calculated from these data. The disagreement in these results is consistent with the observation[3] that physicochemical properties of gelatin systems vary with time and are very sensitive to hydrogen-ion concentration. The values calculated from Lillie's data[4] fall within the range of molecular weights found by these students.

Students' Results

Conc., g./100 ml.	*Height, cm.*	*Mol. wt.*
0.924	4.52	53,500
0.545	4.45	32,000
1.029	3.68	73,000
0.659	5.48	31,300
0.794	2.97	69,700
0.944	3.87	63,700

Despite the simplicity of the experiment, it not only demonstrates the principles of the method, but also shows quite forcefully the magnitude of the molecular weight of a protein and illustrates the application of physicochemical methods to biological problems.

The experiment should be expected to work with other water-soluble proteins such as albumins, possibly with more reproducible results.

[3] GORTNER, R. A., R. A. GORTNER, JR., AND W. A. GORTNER, "Outlines of Biochemistry," 3rd ed., John Wiley & Sons, Inc., New York, 1949, p. 391.

[4] ROBERTSON, T. B., "The Physical Chemistry of Proteins," Longmans, Green and Co., Inc., New York, 1918, p. 338.

THE ART OF SEARCHING THE LITERATURE[1]

I. Search for On-the-Spot Information

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THE National Resources Committee in connection with research has stated: "It has been rather carefully estimated that a thorough study of a problem in a good library, prior to and during the prosecution of research on it, will save on the average 10 per cent of the total cost in time and money." A search serves an investigator, not only in avoiding rediscovery of already known facts, but also as a source of inspiration for germination of fertile ideas. The library can be the first step in discovery, and research should begin in the library. It is in recognition of this fact that colleges are now offering to science seniors courses in the use of chemical literature.

However, searching the literature is an art, in that no precise laws can be laid down, and, given technical training on the part of the searcher (and this is essential) different procedures followed by individuals will arrive at the same result provided methodical thoroughness, persistency, ingenuity, and chemical imagination are used in pursuing the search. A "feel" for the literature must come from working with it. Skill in handling the tools of search, the indexes and reference books, will contribute greatly to speed and efficiency.

Scientific periodical literature, as such, began with the publication of the *Philosophical Transactions of the Royal Society* in 1665. Patent literature began in 1617 with British Patent Specification No. 1. These dates signify transition from Alchemy, with its tomes of "recipes," to Science. We are now in another, and still more prolific era, when industrial research, abandoning its former secrecy, is contributing greatly to scientific advance through technical and trade journal articles, fuller descriptions in patent specifications, and publication of company technical data. Short cuts and better methods in searches are therefore as necessary now as was the mechanization of former manual procedures in the laboratory.

Literature searching may be of several types depending on the purpose: there is (1) search for on-the-spot information, (2) the survey proper which may range from a "scholarly" bibliography, called scholarly in that it is as complete as all printed resources in the world can make it, to mere selected listing of article titles, and (3) the patent search.

Very often a quest for spot information is most time-

[1] Paper presented at the 40th Annual Convention of Special Libraries Association before the Chemistry Section of the Science-Technology Division, June 15, 1949, Los Angeles, California.

consuming, even fruitless, unless the exact directories giving the sought facts are known. The Special Libraries Association has been active in publishing a series of search tools useful to the investigator of scientific literature.[2]

The following lists some general sources for conducting a search for spot information, and some miscellaneous sources mostly in plastics and textiles, the fields with which the author is most familiar. No attempt has been made to be exhaustively complete.

WHAT IS IT?

In the following sources answering the query, "What is it?" a trade name is usually given. Trade name information is widely scattered and none of these compendiums is complete. For that reason a good technical library usually keeps its own card index of trade names encountered in a review of the literature, and this will at least tell which company owns the name, and sometimes the chemical identity of the substance or composition of the mixture covered by the trade name. The glossary of some German names refers to names used in the Office of Publication Reports of German Documents where trade names are often used without any explanation. Rowe's Color Index gives the common name of dyes, their chemical composition, their intermediates and original producer.

General Information

BENNETT, H., "The Chemical Formulary," D. Van Nostrand Co., Inc., New York, **1945,** Vol. VII; **1948,** Vol. VIII (Cumulative Index up to Vol. VI).

"Chemical Industries Buyer's Guide," Trade-Name Section, Maclean-Hunter Publishing Corp., New York (annual publication).

GARDNER, W., "Chemical Synonyms and Trade Names," 5th ed. D. Van Nostrand Co., Inc., New York, **1948.**

GREGORY, T. C., "The Condensed Chemical Dictionary," 3rd ed., Reinhold Publishing Corp., New York, **1942.**

"Thomas' Register," Thomas Publishing Co., Inc., New York (annual publication).

"Trade-Names Index," Special Libraries Association, 31 East 10th St., New York, **1941.**

ZIMMERMAN, O. T., AND I. LAVINE, "Handbook of Material Trade Names," Industrial Research Service, Dover, N. H. **1946.**

Miscellaneous

CURTIS, H. A. (Office of Technical Services), "Glossary of Some German Names for Chemical Products," C.I.O.S. Item No. **22,** Hobart Publishing Co., Inc., Washington, D. C.

"Modern Plastics Encyclopedia," Plastics Catalogue Corp., 122 East 42nd St., New York, (annual publication).

ROWE, F. M., "Color Index," 2nd impression, Mapleton House Publishers, Brooklyn, **1948.**

SIMONDS, H. R., AND C. ELLIS, "Handbook of Plastics," 2nd ed., D. Van Nostrand Co., Inc., New York, **1949.**

"Synthetic detergents commercially available," *Chemical Industries,* **61,** 817–24 (Nov., 1947).

"Textile Brand Names Dictionary," Textile Book Publishers, Inc., New York, **1947.**

[2] LEWTON, L. O., "Contributions of the Special Libraries Association to Searching Technical Literature," presented at the 117th Meeting of the American Chemical Society, Detroit, April 17, 1950.

WHAT IS IT LIKE?

Information on one or all properties of a substance is often desired. The accompanying list furnishes some usual sources. International Critical Tables, difficult as they are to use, refer to original literature. The Rubber Handbook is literally the chemist's "Vademecum." The French "Tables annuelles des constants" has ceased publication, but earlier issues are extant. Beilstein, fourth edition, covers the literature to 1929, but second and third supplements are appearing bridging the gap to 1939. Friedrich Richter, the present editor, hopes, now that access to American literature since 1939 has been given him, to have third supplements to all volumes by 1960. (The Beilstein arrangement and use will be discussed in Part II of this paper.) Elsevier's Encyclopedia, of which so far only five volumes have been issued, promises to be a rival to Beilstein, simpler to use and covering the literature to 1946. Publication started with complex polycyclic compounds; completion is projected by 1960. Simplest to use for ordinary organic compounds is Heilbron's "Dictionary of Organic Compounds," giving the common names as well as the Geneva nomenclature, the compounds being listed in direct dictionary fashion, with ordinary physical constants and derivatives given and a few references to original literature. For solubilities, the second volume of Seidell's "Solubilities of Organic Compounds," still remains the most complete compilation; for inorganic compounds the 16-volume Mellor holds its pre-eminence (there is a one volume abridgement) though Gmelin-Kraut is still the leader in certain respects. Out of print for some time, this classic is now being reproduced both on microcards and in a reprint edition.

Physical

HODGMAN, C. D., *Editor,* "Handbook of Chemistry and Physics," Chemical Rubber Publishing Co., Cleveland, Ohio (annual publication).

LANDOLT-BERNSTEIN, "Physikalische chemische Tabellen," 5th ed., Edwards Brothers, Ann Arbor, Michigan, **1923,** Vols. I and II. 1st, 2nd, and 3rd supplements, reprint **1943.** 6th ed. has been announced.

LANGE, N. A., *Editor,* "Lange's Handbook of Chemistry," Handbook Publishers, Inc., Sandusky, Ohio (annual publication).

LYMAN, T., *Editor,* "Metals Handbook," The American Society for Metals, Cleveland, Ohio, **1948.**

PERRY, J. H., *Editor,* "Chemical Engineers' Handbook," 3rd ed., McGraw-Hill Book Co., Inc., New York, **1950.**

WASHBURN, E. W., *Editor,* "International Critical Tables," McGraw-Hill Book Co., Inc., New York, **1933,** Vols. I–VII and Index.

Chemical

Organic

BEILSTEIN, F. K., "Handbuch der organischen Chemie," 4th edition and 2nd supplements to Vols. 9 and 10, Springer-Verlag, **1949.**

ELSEVIER, "Elsevier's Encyclopedia of Organic Chemistry," Elsevier Publishing Co., Inc., New York, **1948.** Vols. 12A, 12B, 13, and 14 available (polycyclic compounds).

FARADAY, J. E., "Encyclopedia of Hydrocarbon Compounds," Chemical Publishing Co., Brooklyn, N. Y., **1946–50.** Vols. 1–8.

HEILBRON, I. M., "Dictionary of Organic Compounds," Oxford University Press, New York, **1943,** Vols. 1–3.

HUNTRESS, E. H., "A Brief Introduction to the Use of Beilstein's Handbuch der Organischen Chemie," 2nd ed., John Wiley & Sons, Inc., New York, 1938.
SEIDELL, A., "Solubilities of Organic Compounds," 3rd ed., D. Van Nostrand Co., Inc., New York, 1941, Vol. II.

Inorganic

COMEY, A. M., "A Dictionary of Chemical Solubilities—Inorganic," 2nd ed., Macmillan and Co., New York, 1921.
FRIEND, J. N., "A Text-Book of Inorganic Chemistry," C. Griffin & Co., Ltd., London, 1914, 9 vols.
GMELIN-KRAUT, "Handbuch der anorganischen Chemie," 8th ed., Carl Winters Univ. Press, Heidelberg, 1907–15, 5 vols., 12 parts.
JACOBSEN, C. A., "Encyclopedia of Chemical Reactions," Reinhold Publishing Corp., New York, 1946–48, 2 vols. (to Calcium).
MELLOR, J. W., "A Comprehensive Treatise on Inorganic and Theoretical Chemistry," Longmans, Green & Co., New York, 1946–48, 21 vols.
MELLOR, J. W., "Mellor's Modern Inorganic Chemistry," Longmans, Green & Co., New York, 1939, abridgment in 1 vol.

General

BRADY, G. S., "Materials Handbook," 5th ed., McGraw-Hill Book Co., Inc., New York, 1944.
O'ROURKE, C. E., "General Engineering Handbook," 2nd ed., McGraw-Hill Book Co., Inc., New York, 1940.

Textiles and Plastics

"Chart of Synthetic Fibres," *Textile World*, Sept., 1949, McGraw-Hill Publishing Co., New York.
"Modern Plastics Encyclopedia" (chart of properties), Plastics Catalogue Corp., 122 E. 42nd St., New York (annual publication).
SIMONDS, H. R., AND C. ELLIS, "Handbook of Plastics," 2nd ed., D. Van Nostrand Co., Inc., New York, 1949.
"Technical Data on Plastics," Plastics Materials Mfg. Assn., Inc., Tower Building, Washington, D.C., 1948.

WHAT IS IT FOR?

This question leads usually straight to an encyclopedia for a general orientation—Ullman and Thorpe are classics in this field (and include processes, the first one being generally illustrated); the latter work is in the process of revision. Snell's "Chemicals of Commerce" gives briefer information on chemicals only, while Gregory's "Uses and Applications of Chemicals" is most complete on uses, listing them divided by the field, and referring to patents; Kirk and Othmer, the latest addition to encyclopedias, will be more complete and up to date, especially focusing on organic compounds, the articles being written by competent chemists. Many new processes and materials were born during World War II and crept unheralded into industrial use. The U. S. Senate Sub-Committee Monograph No. 2 on Wartime Technologic Developments is a most useful compilation for finding these.

GREGORY, T. C., "Uses and Applications of Chemicals and Related Materials," Reinhold Publishing Corp., New York, 1939, Vol. I, and 1944, Vol. II.
KIRK, R. E. AND D. F. OTHMER, "Encyclopedia of Chemical Technology," Interscience Encyclopedia, Inc., New York, 1947–51, Vol. 1–6 (to Furfural).
SNELL, F. D., AND C. T. SNELL, "Chemicals of Commerce," D. Van Nostrand Co., Inc., New York.
THORPE, J. F., AND M. A. WHITELEY, "Thorpe's Dictionary of Applied Chemistry," 4th ed., Longmans, Green & Co., New York, 1947–49, Vols. I–IX (to Pi).
ULLMAN, F., "Enzyklopadie der technischen Chemie," Urban & Schwarzenberg, 1928–32, Vols. 1–10 and Subject Index. (Reprint, Edwards Brothers, 1943.)
U. S. Senate Committee on Military Affairs, "Wartime Technological Developments," Subcommittee Monograph No. 2, 79th Congress, 1st Session, May, 1945.

WHO MAKES IT?

This is the commonest query. A good technical library should maintain extensive files of manufacturers' catalogues of materials. For equipment Thomas' Register of Manufacturers is the most complete, listing almost all machinery and manufactured products regionally and by subject, as well as giving a rating of size to each firm. Corrosion-resistant equipment is best described in *Chemical Engineering's* tables of "Materials of Construction." One must not forget the advertisements of various manufacturers, especially in annual machinery and equipment issues of magazines, like *Textile World* or Chemical Exposition issues of *Chemical Industries*.

Chemicals

"Chemical Industries Buyers' Guide," Maclean-Hunter Publishing Corp., New York (annual publication).
Oil, Paint & Drug Reporter, "Green Book Buyers Directory," Schnell Publishing Co., Inc., New York (weekly publication).
Catalogs of Eastman, Monsanto, Hercules, Dow, Du Pont, Shell, etc.

Equipment

"Chemical Engineering Catalog," Reinhold Publishing Corp., New York (annual publication).
"Materials of Construction of Chemical Industry," McGraw-Hill Publishing Co., New York.
"Sweet's Machinery Catalog," Sweet's Catalog Service, 119 W. 40th St., New York (annual publication).
"Thomas' Register," Thomas Publishing Co., New York (annual publication).

Miscellaneous

Davison's Textile Catalogs (Knit Goods Trade; Rayon and Silk Trades), Davison Publishing Co., Ridgewood, N. J. (annual publications).
"Textile Chemical Specialty Guide," Rayon Publishing Corp., 303 Fifth Ave., New York (annual publication).
"Directory" (Materials Index), The Society of the Plastics Industry, Inc., 295 Madison Ave., New York (annual publication).
"Modern Plastics Encyclopedia," Plastics Catalogue Corp., 122 East 42nd St., New York (annual publication).
"Check List of Stainless Steels," Peter Frasse & Co., Grand St. and Sixth Ave., New York.
"Organic Finishing Guide-Book," Finishing Publications, Inc., 11 W. 42nd St., New York (annual publication).
"Standard Metal Directory," Atlas Publishing Co., 133 W. 21st St., New York.

HOW MUCH DOES IT COST?

Here are some sources where current prices are best found: *e. g., Oil, Paint & Drug Reporter* lists weekly changes. Monthly prices are given for some chemicals in *Chemical and Engineering News, Chemical Week*, and *Chemical Engineering*.

Chemicals

Oil, Paint & Drug Reporter, Schnell Publishing Co., Inc., New York (weekly publication).
Chemical Week (selected chemicals only), McGraw-Hill Publishing Co., Inc., New York (weekly publication).
Chemical and Engineering News (chemical market prices), American Chemical Society, Washington, D. C. (weekly publication).

Miscellaneous

Black Diamond (for coal prices), Black Diamond Co., Inc., 431 S. Dearborn St., Chicago (bi-weekly publication).
Engineering and Mining Journal (for ore prices), McGraw-Hill Publishing Co., Inc., New York, (monthly publication).
Iron Age (for metal prices), Chilton Co., Inc., Chestnut and 56th Sts., Philadelphia.
Plastics World, Cleworth Publishing Co., 551 Fifth Ave., New York (monthly publication).
Platts Oilgram (for fuel prices), Platts Oilgram, 50 W. 50th St. New York.
Rayon & Synthetic Textiles, Rayon Publishing Corp., 303 Fifth Ave., New York (monthly publication).
Survey of Current Business (for nontechnical commodities), U. S. Dept. of Commerce, Washington, D. C.

HOW MUCH IS THERE OF IT?

Statistical information is most elusive, since figures are often not broken down in the way they are required to be expressed. For instance, no figures are available anywhere on amount of cellulose nitrate used as aeroplane dopes—all information being included under "lacquers, cellulosic." Statistics for organic solvents are well developed both by the government and by the Manufacturing Chemist's Association, which in its "Chemical Facts and Figures" reports over quite a range of years. "World Chemical Developments," of the U. S. Bureau of Foreign and Domestic Commerce, gives a general picture of world production and markets.

General

"Statistical Abstract of the U. S. "(for value of product, size of industry), U. S. Dept. of Commerce, Washington, D. C. (annual publication).
"Biennial Census of Manufacturers," U. S. Dept. of Commerce, Washington, D. C.
"Decennial Census of U. S.," U. S. Dept. of Commerce, Washington, D. C.
"Survey of Current Business," U. S. Dept. of Commerce, Washington, D. C. (annual issue).

Chemicals

Annual review issues of magazines such as *Chemical Engineering* (February issue), and *Paper Trade Journal*.
"Chemical Statistics Directory," U. S. Dept. of Commerce, Washington, D. C.
"Chemical Facts and Figures," 2nd ed., Manufacturing Chemists' Association of the U. S., 608 Woodward Bldg., Washington, D. C., **1946.**
"World Chemical Developments," U. S. Bureau of Foreign and Domestic Commerce (annual publication).

Miscellaneous

"Commodities Yearbook," Commodity Research Bureau, Inc., 82 Beaver St., New York.
"Plastics and Synthetic Resins," U. S. Dept. of Commerce Bureau of Census, Washington, D. C.
Rayon Organon, Textile Economics Bureau, Inc., 10 E. 40th St., New York (monthly publication).
"World Fiber Review," Bulletin No. 14 of Commodity Series, Food and Agriculture Organization of the United Nations, Washington, D. C., August, **1949.**
ROUSH, "Minerals Industries," McGraw-Hill Book Co., Inc., New York, **1892–1941.**
U. S. Bureau of Mines, "Minerals Yearbook," U. S. Govt. Printing Office, Superintendent of Documents, Washington, D. C.

WHAT DOES IT MEAN?

Nomenclature differences, abbreviations, and symbols are true stumbling blocks on the paths of science. More specialized and up-to-date dictionaries are needed. Here some of the latest dictionaries are listed and care must be used in noting the edition, for terminology grows as science progresses. Particularly obscure are textile terms, harking back as they do to the origins of one of the oldest technologies. Plastics terminology is one of the newest and has just achieved in Dickinson's volume its first dictionary.

Foreign words had better be looked up in specialized dictionaries, as a general dictionary may give an entirely different meaning.

Abbreviation and Symbols

"Chemical and Engineering Dictionary," Chemical Publishing Co. of New York, Inc., New York, **1948.**
"Letter Symbols for Chemical Engineering," American Standards Association, Inc., 70 E. 45th St., New York.
ZIMMERMAN, O. T., AND I. LAVINE, "Scientific and Technical Abbreviations, Signs and Symbols," Industrial Research Service, Dover, N. H., **1948.**

Word Glossaries

BENNETT, H., "Concise Chemical & Technical Dictionary," Chemical Publishing Co., Brooklyn, N. Y., **1947.**
DANA, J. D., AND BERMAN, "The System of Mineralogy," John Wiley & Sons, Inc., New York, **1944.**
GOULD, "Gould's Medical Dictionary," 5th ed., The Blakiston Co., Philadelphia, **1945.**
HACKH, "Hackh's Chemical Dictionary," 3rd ed., The Blakiston Co., Philadelphia, **1944.**
MALISOFF, W. M., Editor, "Dictionary of Biochemistry and Related Subjects," Philosophical Library, New York, **1943.**
NEWMARK, M., "Illustrated Technical Dictionary," Philosophical Library, New York, **1944.**
"Van Nostrand's Scientific Encyclopedia," 2nd ed., D. Van Nostrand Co., Inc., New York, **1947.**
WELD, LEROY D., "Glossary of Physics," McGraw-Hill Book Co., Inc., New York.

Textile and Plastics Terms

CARMICHAEL, W. L., LINTON, AND PRICE, "Callaway Textile Dictionary," Callaway Mills, La Grange, Ga., **1947.**
"Dan River's Dictionary of Textile Terms," 5th ed., Dan River Mills, 40 Worth St., New York, **1948.**
MIDGLEY, E., "Technical Terms in the Textile Trade," Emmott & Co., Ltd., 65 King St., Manchester, England, **1931,** Vol. I: "Cloth Terms."
"Rayon Glossary," 2nd ed., American Viscose Corp., 350 Fifth Ave., New York, **1947.**
DICKINSON, T. A., "Plastics Dictionary," Pitman Publishing Corp., New York, **1948.**
"A Glossary of Technical Words Used in Plastics & Rubber Industries," B. F. Goodrich Co., Cleveland, Ohio.

Foreign Language Words

CALLAHAN, L. I., "Russian-English Technical & Chemical Dictionary," John Wiley & Sons, Inc., New York, **1947.**
DEVRIES, L., "German-English Science Dictionary," 2nd ed., McGraw-Hill Book Co., Inc., New York, **1946.**

I. G. Farben. Ind. (Compiler), "Dictionary of Dyeing and Textile Finish Trades (German-English), Dictionaries, Inc., 501 Fifth Ave., New York.

LEIBIGER, O. W., AND I. S. LEIBIGER, "German-English and English-German Dictionary for Scientists," J. W. Edwards, Inc., Ann Arbor, Michigan, 1950.

NEWMARK, M., "Dictionary of Science & Technology in English-French-German-Spanish," The Philosophical Library, New York, 1943.

ONTIVEROS, J. R., "Diccionario Textil Panamericano" (English-Spanish and Spanish-English Dictionary of Technical Terms Used in the Textile Industry), Pan American Publishing Co., Inc., New York, 1949.

PATTERSON, A. M., "A German-English Dictionary for Chemists," 3rd ed., John Wiley & Sons, Inc., New York, 1950.

PATTERSON, A. M., "A French-English Dictionary for Chemists," John Wiley & Sons, Inc., New York, 1921.

SCHLOMANN, A., "Illustrated Technical Dictionary in 6 Languages," Oldenbourg Verlags—A. G., Munich, 1923 (Stechert-Hafner Inc., New York), Vol. 14: "Textile Raw Materials," Vol. 15: "Spinning Terms."

WHO IS HE AND WHERE TO ADDRESS HIM?

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Persons

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HOW SAFE IS IT?

In handling solvents or chemicals, or using them in product formulations to be put on the market, it is often advisable to know the toxicities or physiological reactions which may be produced by the material. The following list shows the common sources for looking up this type of information.

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HAMILTON, A., "Industrial Poisons in the United States," The Macmillan Co., New York, 1925.

LEHMAN, K. B., AND F. FLURY, "Toxicology and Hygiene of Industrial Solvents," The Williams & Wilkins Co., Baltimore, Md., 1943.

"Chemical Safety Data Sheets on Various Toxic Chemicals," Manufacturing Chemists' Association, Inc., 246 Woodward Building, Washington, D. C.

PATTY, F. A. *Editor*, "Industrial Hygiene and Toxicology," Interscience Publishing Co., Inc., New York, 1949, Vols. I and II.

SAPPINGTON, C. O., "Medicolegal Phases of Occupational Diseases," Industrial Health Book Co., Chicago, 1939.

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Another type of question is: "Where to publish a paper?" Aside from the well-known truly scientific journal publications of the various societies, a list of the trade journals with name and address of publisher is given, arranged by subject, in Ulrich's "Directory of Periodicals," R. R. Bowkder Co., New York.

Directories of research awards and fellowships are given in "Scientific and Technical Societies of U. S. and Canada" by the National Research Council, and in the book "American Foundations" by Rich.

This section attempted to suggest sources to hunt up the isolated fact in answering usual queries requiring an answer "on the spot." Longer literature investigations will be discussed in Part II of this paper.

THE LACK OF NATURAL PHILOSOPHY IN OUR EDUCATION[1]

In Memoriam of Wilhelm Ostwald

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FOLLOWING the phrase that "the proof of the pudding is in the eating" (*1*), I made a survey during the last two years embracing a large number of students from liberal arts as well as technical colleges to ascertain how many were aware of the fact that Johann Wolfgang von Goethe (1749–1832) was not only a great poet but also a great natural scientist and philosopher (*10*). The percentage was extremely low, but what was even more disturbing was the fact that about 20 per cent did not even know who this genius of two centuries ago was (*7*).

What is even more deplorable, however, at least from a chemist's point of view, is the fact that not a single one of those approached knew that Wilhelm Ostwald (1853–1932), the pioneer of modern physical chemistry, was also one of the greatest philosophers of his day.

If Wilhelm Ostwald's biography is studied without prejudice or favoritism it must be admitted that his greatest contribution to science and education was not his discovery of how to form oxides of nitrogen by passing a mixture of air and ammonia over a platinum catalyst (a discovery for which he received the Nobel Prize for Chemistry in 1909), but rather the emphasis he always placed in his writings and lectures on the need of the young generation's acquiring at least a basic knowledge of what he called "basic philosophy" during the years it devotes to its education in colleges and universities. When Wilhelm Ostwald visited the United States in the early years of this century, he immediately realized the lack of interest in a philosophical approach in teaching natural sciences in our colleges and universities (*5*, *6*, *9*). To offer proof that he was not the only one to take this attitude one need only refer to a book written twenty years ago by Sir William Dampier Whetham (*11*) in which is found the following statements:

> To the scholar, the man of science sometimes seems to be busy about little facts and trivial problems in an entirely superficial way. On the other hand, to the philosopher or the man of science, if they ignore the underlying verities and look only to literal interpretations, it seems that, as Hume said, "Popular theology has a positive appetite for absurdity." . . . Here again the historical method enables us to get beneath surface trivialities, see the deep secrets of nature that may lie hid in the movements of the needle of a galvanometer or the markings on a butterfly's wing and trace the gropings of man's soul . . . . And so physical science continually widens our knowledge of the phenomena of the natural world and of the relations between the concepts, final or proximate, that we use to interpret the phenomena. On its new lands it builds more temples for the human mind. But also it has dug so deep that before the eyes of this generation it seems to have exposed its foundations and to have reached the unknown ground beneath, which necessarily is of a nature different from that of the superstructure. As Newton said, "The business of natural philosophy is to argue from phenomena . . . and to deduce causes from effects till we come to the very first cause, which certainly is not mechanical."

In the introduction to his book on natural philosophy (*8*), Wilhelm Ostwald has this to say:[2]

> Natural science and natural philosophy are not two fields which oppose each other, but they belong together just as two paths which lead to the same goal. This goal is: The mastery of nature by the human being. This is accomplished by the various natural sciences, all of which collate the actual circumstances between natural phenomena and attempt to discover if and to what extent they depend on each other. This is done in order to be able to predict one phenomenon from what is known of another with more or less accuracy. Natural philosophy joins in such attempts, but only in a more general way. For example, while the science of electricity is a branch of physics which tries to correlate electrical phenomena with other phenomena falling within the realm of physics, natural philosophy is interested not only in correlating all physical phenomena and properties of matter but also its chemical, biological, astronomical phenomena, or, to be more general, all phenomena familiar to us. In other words, natural philosophy is the most general branch of natural science . . . . Every representation of science has its natural philosophical component. In the textbooks, for the lowest as well as the highest grades of education, the natural philosophical chapter can usually be found in the form of an introduction at the beginning or as a general summary at the end of the book. In specific articles in which scientists report their progress, the natural philosophical components are generally present in the form of assumptions, of sentences which are not discussed, frequently not even referred to specifically, but on whose acceptance the most important deductions are based. In all such cases these important sentences can never be found in the right place. If they are contained in the introduction of the textbook they are of little value because the facts they actually should summarize are developed only later in the book. If they are contained in the summary at the end they are too late because they have been previously used already in many instances without, however, referring to their general importance. The best thing to do (and a good teacher will always do it) is to permit generalization

[1] Presented before the Division of History of Chemistry at the 19th Meeting of the American Chemical Society, Boston, Massachusetts, April 1–5, 1951.

[2] Translation of the following quotation was made by E. A. Hauser.

only in such measure as the given individual facts call for and justify.

Therefore, any education in natural sciences is interpenetrated by natural philosophy. How this is accomplished will depend on the way the instructor approaches his problems. Just as one can obtain a survey over a complicated structure, as for example the confusion of streets in a large city, only if one has all information about the individual streets and also knows the relation of one to another after having studied a general city plan, it is advisable when studying a special branch of science to look at the general plan so that one does not become confused whenever one's path leads through an unfamiliar section.

To accomplish this broader outlook, courses in the history of science and technology, and particularly of natural sciences, should be made compulsory subjects for every student in liberal arts and technological colleges and universities. Such courses should never be substitutes for courses in any specific branch of natural science but should offer every student a chance to learn the importance of natural philosophy for his future. The best way to accomplish this would be to offer the student as vivid a picture as possible of such men as Michael Faraday and Richard Zsigmondy, to mention only two. By explaining to the student how these men made their greatest contributions to science it will be easy to imbue him with the basis of natural philosophy and to show him how he can apply this most important branch of natural science for his own good. To prove how this could be done, the following quotations might be of interest.

When Michael Faraday presented the Bakerian Lecture in 1857 (*3*) he made the following statements:

The particles in these fluids [he was referring to fluids containing gold and how they appear when a concentrated beam of light is passed through them] are remarkable for a set of physical alterations occasioned by bodies in small quantities, which do not act chemically on the gold, or change its intrinsic nature; for through all of them it seems to remain gold in a fine state of division. They occur most readily where the particles are finest, i.e., in the ruby fluids .... An idea of their nature may be obtained in the following manner. Place a layer of ruby fluid in a clean white plate, dip the tip of a glass rod in a solution of common salt and touch the ruby fluid; in a few moments the fluid will become blue or violet-blue.

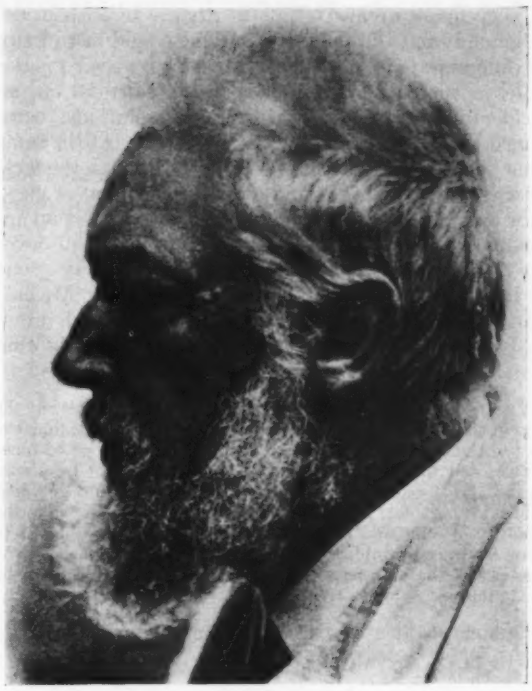
Such results would seem to show that this blue gold is aggregated gold, *i. e.*, gold in larger particles than before, since they precipitate through the fluid in a time which is as nothing to that required by the particles of the ruby fluid from which they are obtained. But that the blue particles are always merely larger particles does not seem admissible for a moment, inasmuch as violet or blue fluids may be obtained in which the particles will remain in suspension as long as in the ruby fluids; there is probably some physical change in the condition of the particles, caused by the presence of the salt and such affecting media, which is not a change of the gold as gold, but rather a change of the relation of the surface of the particles to the surrounding medium.

When salt is added in such quantity as to produce its effect in a short time, it is seen that the gold reflexion of the particles is quickly diminished, so that either as a general turbidness or by the cone or rays it becomes less visible; at last the metal contracts into masses, which are comparatively so few and separate, that when shaken up in the fluid, they confer little or no colour or character, either by reflected or transmitted light ....

Many other bodies besides salt have like action on the particles of gold. A ruby fluid is changed to or towards blue by solutions of chlorides of calcium, strontium, manganese; ...

Amongst the alkalies, potash produces a similar action in a weak degree .... Lime-water produces a change in the same direction, but the gold quickly precipitates associated with the lime ....

The state of division of these particles must be extreme; they have not as yet been seen by any power of the microscope. Whether those that are ruby have their colour dependent upon a particular degree of division, or generally upon their being under a certain size, or whether it is consequent in part upon some other condition of the particles, is doubtful (*2, 4, 5, 6*).



Wilhelm Ostwald

I picked this citation for the following reason. In discussing the importance of natural philosophy in our education with Wo. Ostwald, one of Wilhelm Ostwald's sons, when he visited the United States for the last time in 1938, he made the following statement: "When my book, 'The World of Neglected Dimensions,' appeared in print in 1915, my father criticized it for the lack of natural philosophy. Referring specifically to Faraday's observations he felt that all problems pertaining to the electrokinetic properties of colloidal sols would have been solved long ago if more attention had been paid to the history of science and to natural philosophy combined therewith."

One of the most striking proofs of the need of natural philosophy in our education is to be found in a historical review of Richard Zsigmondy's discovery of the ultramicroscope. He was one of the strongest supporters of the so-called "allotropic" or "solution" theory, which stated that there is no satisfactory reason for imagining the existence of a sharp boundary between a solution and what Thomas Graham called a colloidal sol. In contrast thereto the "heterogeneous" theory, of which Wilhelm Ostwald was one of the strongest supporters, assumed that colloidal solutions are composed

of very finely divided solids in a state of suspension. Zsigmondy felt that since these fluids could in practice be differentiated from each other only by their greater or lesser turbidity, turbidity is not an essential but an incidental characteristic of colloidal solutions, occasioned by the presence of larger particles. At the same time he acknowledged, however, that although the facts referred to seemed to be favorable for his point of view, they actually gave no evidence of the existence of an optically clear solution. He hoped to be able to discover the truth by microscopic examination of Faraday's cone of light. This was an observation made by Michael Faraday at the same time as those mentioned previously; referring to a ruby fluid he made the following statement (*3*):

> The particles are easily rendered evident, by gathering the rays of the sun (or a lamp) into a cone by a lens, and sending the part of the cone near the focus into the fluid; the cone becomes visible, and though the illuminated particles cannot be distinguished because of their minuteness, yet the light they reflect is golden in character, and seen to be abundant in proportion to the quantity of solid gold present. Portions of fluid so dilute as to show no trace of gold, by colour or appearance, can have the presence of the diffused solid particles rendered evident by the sun in this way.

Zsigmondy's idea was based on the following reasoning (*12*):

> It seemed to me probable that the larger particles assumed to be present in gold hydrosols, whose previously mentioned property of dispersing transmitted light was known to me by the above experiments, could be made individually perceptible by a microscopic examination of the light cone; for if the small particles reflected enough sunlight, even if their size was below the limit of microscopic resolvability, they would, just as were the narrow rays of light, be individually perceptible under the microscope and act to a certain extent as fragments of such light slits. The microscopical investigation should also allow me to determine if the space between the coarser individual particles was optically clear or filled with smaller gold particles.

When Zsigmondy then made his first observations he had to admit that his theory was based on assumptions only and not on experimental facts. He pointed out to me personally that he owed this discovery largely to the teachings of Wilhelm Ostwald of the importance natural philosophy should play in the life of every scientist, and although he had originally opposed Ostwald's theory, he had become so imbued with his teachings that he decided that only a logical and philosophical approach to the problem would offer the facts needed to arrive at a decision as to which of the two opposing assumptions was correct.

I doubt if I could offer any better proof of what a historical and natural philosophical approach to science would mean in our education than to quote from James B. Conant's most recent book (*2*):

> With idolatry of science I must confess I have little sympathy. Yet a better understanding of the methods of the natural sciences among laymen is certainly to be desired. Since scientific investigations provide wide spread and often dramatic examples of an effective way of handling problems, a greater knowledge of the genesis of scientific methods ought to be imparted by our schools and colleges.

Since Wilhelm Ostwald was beyond any question of a doubt the first to put all the emphasis at his disposal on the part natural philosophy should play in our education, I consider it only right to dedicate this paper to the memory of one of the greatest scientists who ever lived.

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Even photography is becoming more and more closely associated with electrical processes. A new camera which produces finished pictures in two minutes uses an electrostatic-electrophotographic process. Light is recorded as an invisible electrical image on a selenium coated 4 by 5 in. metal plate. The plate is first sensitized by an electrical charge. Fine powder dusted over the plate is held by the charge. This powder image is then transferred to an adhesive coated paper or other material.

THE POLAROGRAPHIC DIFFUSION CURRENT AND THE HEIGHT OF THE MERCURY RESERVOIR

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THE principle of zero suppression[1] is well known in polarography, but the following may be a new and useful application. The results obtained more than justify the extra complications introduced.

In the experimental verification of the relation $i_d \alpha \sqrt{h}$, where i_d is diffusion current and h is height of mercury reservoir, a potential of one volt is applied to a polarographic cell containing an air-free $M/1000$ cadmium solution in $M/10$ potassium chloride ($E^1/_2$ = 0.7 v.), and the variation of i_d, due to deposition of Cd^{++}, with h studied.[2]

The above method suffers from the disadvantage that the increase of i_d only amounts to about 20 per cent of the initial current, so that changes in galvanometer readings are relatively small and difficult to read accurately.

A typical plot obtained by students using this method is shown in Figure 1 (Line I).

To increase the accuracy of the experiment the following procedure was adopted.

[1] LINGANE, J. J., AND H. KIRLINGER, *Ind. Eng. Chem., Anal. Ed.*, 12, 750 (1940).
[2] MÜLLER, O. H., J. CHEM. EDUC., 18, 172–7 (1941).

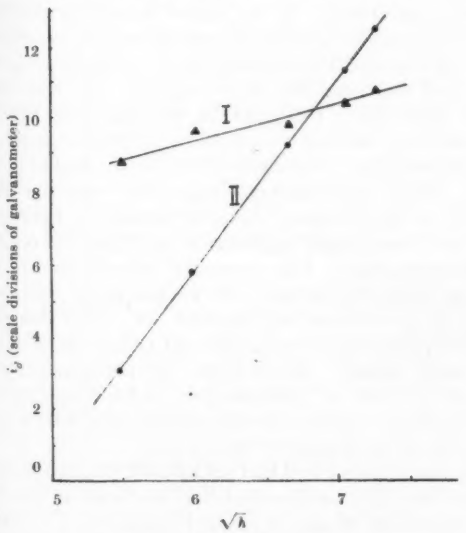

Figure 1
I = Conventional method. II = Modified method.

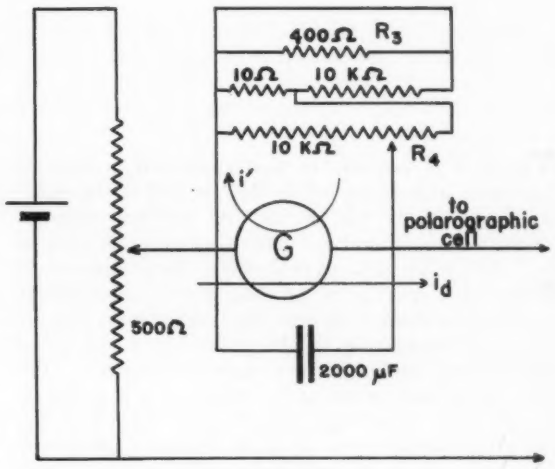

Figure 2

The usual galvanometer shunt was removed and the circuit shown in Figure 2 arranged so that by adjustment of the potentiometer R_4 a small current, i', could be sent through the galvanometer in the opposite direction to that flowing in the polarizing circuit, and this small current, i', was arranged to be equal and opposite to that flowing at the lowest height of the mercury reservoir—that is, to return the galvanometer spot to zero. As this small opposing current must remain constant, resistance, R_3, was connected to the accumulator to act as a load and steady the voltage.

Owing to removal of the shunt the galvanometer was now much more sensitive, and an increase in height of the reservoir caused a much larger deflection of the galvanometer spot. Consequently, the change of i_d with h could be determined accurately (Line II in Figure 1).

The increase in sensitivity of the galvanometer increased the variations in current during drop formation, but this did not affect the accuracy since "average" values for i_d were determined after noting the minimum and maximum readings of the galvanometer at any one height.

A Cambridge galvanometer with a resistance of 450 ohms and a full-scale deflection for 1.5 microamperes was used in these experiments.

Proceedings of the

PACIFIC SOUTHWEST ASSOCIATION OF CHEMISTRY TEACHERS

THE POWER OF MICROANALYSIS[1]

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WHILE it is possible to find analytical procedures which were in use long before the last half of the eighteenth century, it was during the exciting years of Priestley, Scheele, and Lavoisier that *analysis* came of age. As a basis of analytical work, the acceptance of the law of conservation of mass was a prerequisite. Lavoisier, the French chemist, studied the mass relation in chemical changes by weighing starting materials and products, and made the following clear statement: "The whole art of experimenting in chemistry is founded on this principle: in all experiments one is obliged to suppose a true equality, or equation, between the principles of the substances which one examines and those which one obtains by analysis." With these words he introduced the analytical balance as an indispensable tool for generations of chemists.

On the basis of careful analysis the laws of constant composition, and multiple proportion and Avogadro's hypothesis were expounded, and as a direct result was published "A New System of Chemical Philosophy" (Dalton, 1808)—the atomic theory. Once the procedure of weighing before and after a reaction and of accounting for the products formed was discovered, rapid progress was made in the analysis of chemical compounds, leading to the enormous amount of chemical data collected in nine feet of Beilstein's "Handbook of Organic Chemistry" and six feet of Gmelin and Kraut's "Handbook of Inorganic Chemistry."

It is interesting to read of these historical analytical procedures and to contrast them with our present-day techniques. In the early work of Gay-Lussac, half a gram of material mixed with $KClO_3$ was burned in a vertical combustion tube. The $KClO_3$ was replaced in 1818 by Liebig, who introduced the horizontal tube filled with CuO, using absorption apparatus for CO_2 and H_2O. Although his contemporaries were not impressed, this method has been unaltered in principle for over 100 years.

In the type of problem attacked during the last century, material was sufficiently abundant that the analytical methods could be applied where from 200 to 300 mg. of substance was used for the analysis. However, in the investigation of natural products it must have occurred that the products obtained from degradation were so small that no attempts were made to determine their composition. Most of the investigators probably set aside their small amounts of isolated material for future study, as is the habit of quite a number of chemists, even today. There was, however, one chemist, F. Pregl, who was not so easily defeated, and decided that his investigations should not be hampered by limitations in the existing analytical methods.

In 1910, at the University of Innsbruck in Austria, Pregl was studying the degradation of cholic acids. After isolating desoxycholic acid from ox bile, oxidation with HNO_3 had yielded small amounts of crystalline oxidation products. In the paper describing the degradation we notice several remarks about the expensiveness and the small quantities of the breakdown products, and the need for repeating the reactions with larger quantities. Using 200 to 300 mg. for his analysis must have seemed such a waste to Pregl that he decided to find analytical methods for much smaller quantities. These new methods were first reported at a meeting of the German Chemical Society in February, 1911, and found rapid application in all fields of organic and biochemistry. The successful introduction of the new methods was largely due to the great care with which the procedures were worked out. Ten thousand precision weighings were carried out before the methods were made public. In addition, the personal instruction of hundreds of microanalysts in his laboratory in Graz created a nucleus of well-trained men who spread the methods to all parts of the world.

The task which Pregl had set himself was so fascinating and kept him so absorbed that he never returned to his old problem of the cholic acid degradation. Others like Windaus and Wieland carried on these investigations, taking full advantage of the opportunities of the new analysis of milligram quantities, and laid the foundation for the structures which we recognize in numerous

[1] Presented in the PSACT program at the Pacific Southwest Regional Meeting of the American Chemical Society, Whittier College, Whittier, California, April 28, 1951.

[2] At present, Chief Research Consultant of the Los Angeles Air Pollution Control District.

natural products such as sterols, sex hormones, pituitary hormones, and saponins.

At the present time the majority of the organic analyses reported in the scientific journals are carried out according to Pregl. Hand in hand with the refinement of the analyses went the development of microtechniques of recrystallization, fractionation, and other laboratory procedures. Degradations were selected and carried out in such a way that high yields of the small quantities could be expected. Since the success of microanalysis is so largely dependent on recrystallization, we notice a great deal of effort in the characterization of compounds with derivatives which have desirable solubility, properties, and melting points. Fatty acids, for example, which by themselves are not easily characterized in microquantities because most of them are liquids, can be converted into anilides, *p*-nitrobenzoyl, and substituted phenylphenacyl derivatives of excellent crystallization properties. In a similar fashion, aldehydes react with substituted phenylhydrazines, giving beautifully crystallized hydrazones. The introduction of the halogen or nitrogen into the molecule, in conjunction with a micro Rast molecular weight determination, permits the accurate determination of the number of the hydrazines which have reacted with the carbonyl groups. In addition, the increase in weight of the molecule through the introduction of the large molecules of the reagent facilitates the handling of the compound. The introduction of atoms other than C and H is an aid in assigning the correct empirical formula to the compound.

It is often necessary to distinguish between formulas which are different by only one CH_2 group by means of carbon and hydrogen analysis. This is practically impossible for hydrocarbons of the general formula C_nH_{2n+2} but as soon as additional atoms other than C are introduced, the differences between C_nH_{2n+2} and its homologues become detectable in the analysis. A maximum in this difference is reached when the weight of the foreign atoms reaches that of the carbon. For example, the difference between the carbon values of $C_{15}H_{32}$ and $C_{14}H_{30}$ is only 0.1 per cent, whereas that of $C_{15}H_{30}Br_2$ and $C_{14}H_{28}Br_2$ is 1.3 per cent, far within the 0.2 to 0.3 per cent limit of accuracy of the micro CH determination.

With the introduction of chromatographic procedures, the use of derivatives has found a new application. Absorption of the colored nitrophenylhydrazones, fluorescing phenylphenacyl derivatives of acids, makes it possible to purify small quantities of material.

The reseach chemist who, after months of work purifying tons of material, must offer a few per cent of his expensive crystals to the analytical chemist exerts some pressure toward the use of still smaller quantities. From the macrogram analysis we have gone through microgram, and even to submicrogram scale. The sample size of 100 mg. in the macro methods has been reduced to 10^{-5} to 10^{-6} mg. Dr. Kirk lists the scales of the existing micro methods in his book on quantitative ultramicroanalysis as follows (*8*):

Scale of Micro Methods

Method	*Sample size, mg.*	*Reduction from macro*
Macro	100	.....
Semimicro	10–20	$1/5$–$1/10$
Micro (milligram)	1	$1/100$
Ultramicro (microgram)	0.001	$1/100{,}000$
Submicrogram	0.000001–0.00001	10^{-7}–10^{-8}

The ultramicro methods find special application in the field of biology when no isolation of the pure substance is necessary. No procedure comparable to the Pregl gravimetric methods has so far been invented. One of the difficulties was the lack of suitable balances. However, with the introduction of the quartz fiber balance capable of weighing rapidly and reliably to ± 0.005 μg. and a load of several tenths of a milligram, gravimetric methods may be developed, although it should be realized that the Pregl method cannot be adapted to the microgram procedures without major changes in the combustion and sampling procedures.

The most important progress in microgram analysis has been made in the titrimetric and colorimetric procedures. It is possible to carry out routine micro Kjeldahl determinations of NH_3 in which the quantities of nitrogen are in the order of 0.5 μg. with an error rarely exceeding 1 per cent. (Brüel, *et al.*—0.1 μg. with limiting sensitivity of 0.005 (*2*).) The important feature of this and similar methods is that no transfer of the solution is necessary, and digestion and distillation are carried out in the same vessel. The steam distillation of the ordinary Kjeldahl is replaced by a diffusion of the liberated NH_3 into a cup holding the standard acid solution. In this isothermic distillation, at room temperature, the ammonia is quantitatively diffused in 70 minutes.

The advantages of the microgram methods are obvious in blood analysis, where minute samples as small as 0.01 ml. suffice for carrying out a quantitative analysis of reducing sugars with a reproducibility of 1 per cent or better. In this case, a deproteinization of the blood with copper sulfate and sodium tungstate solution is carried out in a centrifuge tube not much bigger than 0.1 ml. To an aliquot of this deproteinized blood is added ferricyanide solution and the reduced iron is titrated with .01 *N* ceric sulfate taken from a capillary buret (*9*). Other fields of application are in the isolation of radioactive compounds where the reduction of quantities in microgram analysis reduced the danger of handling highly active materials to a safe level.

The microanalyst has realized that the division in science such as physics, chemistry, and biology is an artificial one, and that these self-imposed barriers should not prevent his looking to other fields for inspiration to solve his problems. At the present time biological methods are standard practice for the determination of minute amounts of substances (*5*). Whenever a substance is known to have a physiological effect, this effect can serve as a measure of the amounts of material

involved. This type of measurement has been extensively used in the hormone and vitamin field, and was extended to the analysis of amino acids with the help of microorganisms.

Advantage is taken of a nutritive deficiency created by leaving out of the nutrient medium essential amino acids. The organism responds to the addition of small amounts of the lacking amino acid with a growth response, which in the case of the lactobacillus bacteria can be measured by titrating the acid formed from the glucose of the medium. This assay readily takes its place in the microgram scale of analysis, since often 0.1 μg. of amino acid can be determined with an accuracy of a few per cent. For nearly all of the naturally occurring amino acids microbiological analytical methods have been worked out, and these are at present an indispensable tool in the study of protein composition.

The intense interest developed in the last few years in the analysis of amino acid mixtures has given rise to two other methods, partition chromatography (*13*) and the countercurrent distribution method (*4*). Both methods are suitable for the determination of the amino acids in only a few milligrams of protein hydrolysate. In partition chromatography the amino acid mixture is dissolved in butanol, propanol, and HCl solvent and passed through a column of wet starch. According to the speed with which the individual amino acids are moving through the column, they can be collected and determined colorimetrically using the ninhydrin reaction. Whereas in this method the adsorption and partition between the nonmiscible liquids determine the order in which the compounds are passing through the column, the countercurrent distribution method is based solely on the partition between two immiscible solvents. In principle, the method consists of many separatory funnels which are so ingeniously arranged that by a single turn of the apparatus the top layer moves from one separatory unit to the other, carrying with it the extracted material. Some 220 of these units repeat the procedure, and at the end of the run the individual substances have moved through the system with a speed directly related to their partition coefficients. After this separation, colorimetric or other analytical determinations will give the amount of substance present in each fraction.

Chromatographic methods on columns and on filter paper have proved to be of great value in many problems other than amino acid analysis. Sugars, alkaloids, terpenes, plant pigments—in short, in all branches of chemistry—the method is being applied for the separation of small quantities of material. The solution of difficult problems such as that of photosynthesis has received a great stimulation through the combination of isotope and chromatographic techniques. It has further become one of the best criteria of purity of the compounds to be analyzed.

Notwithstanding the choice of separation and analytical techniques, the field of hydrocarbons was most carefully avoided by most microchemists, and it is only in the last few years that satisfactory microanalytical procedures have been developed by the physicist. Infrared (*7*) and mass spectroscopy (*12*) have been shown to be indispensable in these problems. Extremely small amounts of mixtures of saturated as well as unsaturated hydrocarbons can be analyzed with an accuracy of a few per cent. In the case of the mass spectrograph, the masses of the charged fragments of the molecules formed by electron impact are determined. The known patterns for the pure materials allow a computation of the amounts present in the mixture. Recently we had the opportunity to appreciate the possibilities of the mass spectrograph in a study of the air pollutants in the Los Angeles area. The main pollutants of this atmosphere are hydrocarbons, and Dr. M. Shepherd was able to identify from the mass spectrographic data several dozen hydrocarbons present in only two cubic feet of air.

Interesting contributions to microanalysis have been made by X-ray diffraction methods. Well known are the revolutionary results obtained from the investigation of steroids by this method, when Bernal and Crowfoot (*1*) were able to overthrow the old formula. The structure established by the chemists through many years of painstaking work had to be drastically revised. A similar contribution of X-ray analysis has appeared in the structural analysis of penicillin, where a decision could be made between the oxazolone and the β-lactam formula for penicillinic acid (*3*). These spectacular contributions were made from data on single crystals, weighing often not more than 10^{-6} g. Molecular weights, for which the Rast micro method requires 0.1 mg., can be determined with great accuracy using quantities one hundred times smaller.

With all these exciting discoveries in analytical procedures, it is remarkable that there is a widespread notion among fellow scientists that analysts are a rather dull and unimaginative group of people. This attitude is reflected in a lack of prestige and monetary reward. The American Chemical Society was rightly concerned about this situation, and editorials discussing the profession of analytical chemistry have appeared in its journals (*10*). In one of these W. J. Murphy pointed out that the analytical chemist is a mighty important individual, making possible new industries and new processes, protecting the manufacturer's reputation regarding the quality of both raw materials and finished products, frequently making possible the upgrading of old products, and helping to eliminate wastes. The analytical chemist is the guardian of the nation's health and helps to raise the health standards. This, by itself, should remove any idea that the analytical department is a necessary evil, a nuisance that cannot be eliminated, of course, but one which is just dead weight expense. In any research problem the analytical part holds a key position to success.

The widespread introduction of instrumentation has caused a sharp division in the analytical laboratory between those of professional and subprofessional training, experience, and ability. Today thousands of analytical procedures are carried out readily by laboratory

technicians. The true professional is expected to direct, to administer, and to pioneer research in analytical chemistry. He is therefore required to be an organic chemist, an inorganic chemist, a physical chemist, and may, at times, be expected to be a biochemist, a metallurgist—a specialist, if you will, in a dozen or more highly specialized fields. He most certainly must be somewhat of an expert in electronics; he must be almost as much a physicist as physicists themselves. In addition, he is usually expected to be specially skilled in some field within the profession of analytical chemistry.

After describing this ideal analytical chemist who has become a superman among his fellow scientists, several recommendations were made by Mr. Murphy which could improve the analyst's position. I consider as one of the most important of these recommendations the establishment at the universities of a curriculum for a doctorate in analytical science. One field of specialization should be chosen, but in addition the training should be so broad that the student has the necessary background to be aware of the developments in other fields, and to apply these advances to the problems facing him.

The widespread use of microchemistry in all branches of chemistry and biology has made the microanalytical chemist of the past the analytical chemist of today, and the training of the chemist should be directed to keep abreast of this trend in the field of analytical chemistry.

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ANNOUNCEMENT

The Southern California Section of the Association will hold a meeting on Saturday, September 29, at Scripps Institution of Oceanography in La Jolla.

In addition to the excellent climate and natural beauty of the region, La Jolla and neighboring San Diego have much to offer visitors in the way of sightseeing and recreational facilities. It is hoped that members and guests of the Section will plan to spend the week-end in the San Diego area.

Lionel Joseph of San Diego State College is in charge of arrangements.

To the Editor:

In the August, 1951, issue of This Journal there appeared an article written by me entitled "Large crystals of monoclinic sulfur." The method, in brief, was to dissolve the sulfur in olive oil at a temperature of 130°C. and then very slowly to allow the mixture to cool to room temperature in a thermos bottle. Crystals of monoclinic sulfur up to 1 in. in length were obtained.

Working along the same lines I tried to apply this method to selenium. I reasoned that there was a fair likelihood of success since selenium and sulfur were in the same family of elements.

In a measure the results were disappointing. I did not produce large size crystals of selenium but made a new discovery which appears to be of more scientific interest.

It took but a few minutes of heating 1 g. of selenium in 25 ml. of olive oil to note that selenium, unlike sulfur, was not appreciably soluble in olive oil even though the oil was heated to its boiling point. I put aside the test tube and contents and hoped to try some new solvent some other day. The next morning I was

astonished when I observed that this oil had been transformed into a fatty crystalline ivory-white solid, which when analyzed was found to contain about 0.2 per cent selenium. Thus the selenium appears to act as a catalyst.

I am not in the position to determine the composition of this substance and would welcome possible guidance from the readers of THIS JOURNAL. Cottonseed and corn oils when treated in the same manner did not solidify upon standing.

MILTON G. WOLF

JAMES MADISON HIGH SCHOOL
BROOKLYN, NEW YORK

To the Editor:

We have noted with interest the article "Oil and water emulsions: a lecture demonstration" by Sister M. Ignatia (J. CHEM. EDUC., **28,** 112 1951). It appears to the writer that the clarity of the demonstration might be improved in the following manner:

In the demonstration cited a single formulation is used to prepare both O/W and W/O emulsions, the type of emulsion depending upon the method of preparation. It is possible that a mistaken conclusion might be drawn to the effect that the method and order of mixing determine the nature of the product. The type of emulsion usually depends more upon the hydrophile-lipophile balance of the emulsifier rather than on the method of mixing the ingredients.

Use of the Waring Blendor in the preparation of these emulsions is inadvisable because it is possible to instill the belief that such violent agitation is necessary in preparing a satisfactory emulsion.

We suggest the following formulations for such a demonstration:

For O/W Emulsions

Mineral oil (Nujol)	100 ml.
Span[1] 40 emulsifier (molten)	8 ml.
Tween[1] 40 emulsifier	8 ml.
Water	200 ml.

To 100 ml. of oil in a 400-ml. beaker add 8 ml. of molten Span 40 and 8 ml. of Tween 40. Warm the oil and emulsifiers to 50°C. Stir by hand with a spatula until the emulsifiers are uniformly dispersed in the oil. Add water in small increments, stirring thoroughly after each addition. As the water is added, the emulsion (W/O at first) will become thicker and after about 80–100 ml. of water have been added, the maximum viscosity will be achieved. Further addition will cause a sudden reduction of viscosity. At this point, the emulsion has inverted to the O/W type. The balance of the 200 ml. of water may be added rapidly. Preparation by this "inversion" technique *usually* produces a finer particle size and a more stable O/W emulsion than by adding the oil to the water. To obtain good inversion the emulsifier must be properly balanced. In this example, the emulsifier has reduced the work required, violent agitation consequently being unnecessary.

An example of an O/W emulsion as presented in the last paragraph of the article (containing a small amount, 0.5 per cent emulsifier, when the work must be supplied mechanically) is also of interest in comparison with the above formula. When it is desirable to use only a minute quantity of emulsifier a formula such as 20 ml. of water, 0.5 g. of Dreft (or 0.5 ml. of Tween 80), and 80 ml. of oil may be employed. With a small amount of emulsifier a Waring Blendor, homogenizer, or colloid mill must be employed.

For W/O Emulsions

Mineral oil (Nujol) or petrolatum	100 ml.
Span 80 emulsifier	15 ml.
Tween 80 emulsifier	1 ml.
Water	200 ml.

To 100 ml. of oil or petrolatum in a 400-ml. beaker add 15 ml. of Span 80 and 1 ml. of Tween 80. Stir by hand with a spatula until the emulsifiers are uniformly dispersed in the oil. Add water in increments, stirring throughly after each addition. The emulsion will increase in viscosity as the amount of water is increased and at 200 ml. of water, the emulsion has a heavy "body." However, it will not invert, and if water addition is carried beyond "saturation" of the W/O emulsion, the emulsion will break. To obtain a good commercial W/O emulsion, the product obtained after the addition of the 200 ml. of water should be passed through a homogenizer.

We believe that the above tests will present a more accurate demonstration of the action of emulsifiers.

WILLIAM C. GRIFFIN

ATLAS POWDER COMPANY
WILMINGTON, DELAWARE

[1] Reg., U. S. Pat. Off.

To the Editor:

The article entitled "As I knew Michael Faraday" by James R. Irving which appeared in the June issue of the JOURNAL OF CHEMICAL EDUCATION brings to mind a Faraday letter which I donated to the Library of the Chemists' Club, New York City, a short while ago. This is addressed to H. Hopwood, Esq., and reads as follows:

R. Institution
June 17

Sir

I have not time to go into your objection to the atomic theory. It would require a view of what we know of chemical affinity but you will find the difficulty referred to in most accounts of the atomic theory especially in the earlier accounts.

I do not myself adopt the term atomic theory or atom. I do not know what an atom is. The theory of definite proportionals seems to me consistent and being the expression of a multitude of facts is as relates to these facts, quite true. Whether it be or be not universal is of little consequence to the multitude of useful cases in which it does apply.

The hypothetical views which under the term atomic theory

have been founded on the theory of definite proportionals I have always held as of very suspicious character.

M. Faraday

H. Hopwood Esq.

Of course, Faraday's feelings about the atomic theory are rather well known. In J. H. Gladstone's biography of Faraday, published by Harper and Brothers in 1872, the following appears:

. . . in conceiving of atoms, he (Faraday) says: "As to the little solid particles . . . I can not form any idea of them apart from the forces, so I neither admit nor deny them. They do not afford me the least help in my endeavor to form an idea of a particle of matter. On the contrary, they greatly embarrass me."

I felt that the publication of these items was timely in view of Mr. Irving's article and the tremendous interest in atoms and atomic energy existent today.

RAYMOND SZYMANOWITZ

ACHESON COLLOIDS CORPORATION
PORT HURON, MICHIGAN

Recent Books

• AMINOPLASTICS

C. P. Vale, British Industrial Plastics Ltd. Interscience Publishers, Inc., New York, 1950. 250 pp. 19 figs. 26 tables. 13 × 19 cm. $2.75.

AMINOPLASTICS (or aminoplasts) are defined as resinous products resulting from the interaction of an aldehyde (usually formaldehyde) with compounds containing more than one $—NH_2$ group per molecule. Commercially important members of this class are urea, melamine, thiourea, and guanidine.

The purpose of this book is to collate the technical literature relating to these resins and to their various industrial applications. The first three chapters review the chemistry of the amino resins. Two chapters are devoted to molding powders, processes, and products, followed by a chapter on the physical properties of aminoplastic moldings. The three remaining chapters are concerned with the applications of aminoplastics in lacquers, in adhesives, and laminating resins, and in resins for the textile and paper industries. A list of pertinent references is provided at the end of each chapter.

The book is well written by a highly qualified authority in this field. His discussions of the formation and structure of the aminoplasts are excellent and clarify a rather confused literature on these topics; his chapters on molding powders and other applications are somewhat less satisfying and are not sufficiently complete to serve as more than a very general guide to practical operations.

As a small volume of therefore limited scope and modest price, this book can be recommended as a valuable contribution to the technical literature.

H. A. NEVILLE

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BETHLEHEM, PENNSYLVANIA

• METHODS AND MATERIALS FOR TEACHING GENERAL AND PHYSICAL SCIENCE

John S. Richardson and *G. P. Cahoon,* Department of Education, The Ohio State University. McGraw-Hill Book Co., Inc., New York, 1951. viii + 485 pp. 547 figs. 16 × 24 cm. $4.50.

ANY teacher starting out in the field of general or physical science at the high school and elementary college level will find this book invaluable. It will save many hours of time and pep up the classroom demonstrations. In Part One there is a general discussion showing how demonstration techniques can be used most effectively. The authors warn against experiments which are merely spectacular. They cover the use of lecture demonstrations, projects and reports, field trips, laboratory experiments, and visual and auditory materials. There are a number of plans for demonstration apparatus that can be built with a minimum of skill and shop facilities. A long chapter covers the care of equipment.

Part Two (about two-thirds of the book) is devoted to suggested demonstration experiments in general science, physics, and chemistry. There is a wealth of material to choose from, the experiments are practical and really show something, and most of them require very simple equipment. The beginning teacher will find the numerous line drawings helpful.

It takes a great deal of time and ingenuity to work up a good repertoire of demonstration experiments yet no good teacher would let his class down by leaving them out. Richardson and Cahoon have done us all a service by assembling this material which has something new for the most experienced of us.

RICHARD WISTAR

MILLS COLLEGE
OAKLAND, CALIFORNIA

• CHEMICAL KINETICS

Keith J. Laidler, Associate Professor of Chemistry, The Catholic University of America. McGraw-Hill Book Co., Inc., New York, 1950. ix + 408 pp. 44 figs. 51 tables. 15 × 23.5 cm. $5.50.

PROFESSOR LAIDLER'S new book deserves the simple but inclusive title he has chosen, including as it does a wealth of material on every important aspect of chemical kinetics. It is primarily intended as an introductory text for students of graduate level, but is useful as well as a reference book because of its rather complete coverage of the literature in the field.

Chemical kinetics may be divided in two parts, the kinetics of elementary reactions and the kinetics of complex reactions. Professor Laidler discusses the first in Chapters 1 to 6 and 13, taking up in order fundamental concepts and definitions, experimental methods, and general theory and mechanisms, and then elementary reactions in the gas phase, in solution, on surfaces, and in the solid state. A very good treatment of the elements of the absolute reaction rate theory is included.

Chapters 7 to 12 are concerned with complex reactions in general, in which the author attempts with good success to show how complex kinetics are related to the simpler kinetics, how complex mechanisms result from combination of simpler mechanisms under various conditions. He stresses not the differences but the similarities between reaction types, which seems a good idea in a textbook. In this section are chapters on general complex reac-

tions, both homogeneous and heterogeneous, complex inorganic and organic reactions, catalysis, oxidation, and polymerization. A great deal of descriptive material is included, but the basic approach is theoretical; the author demonstrates very well the use of experimental kinetic and thermodynamic data in evaluating proposed mechanisms for these reactions. Recent work in several specific fields such as photochemistry, radiochemistry, biological kinetics, and catalysis is included.

Chapter 13 is a very useful discussion of potential energy surfaces for reactions, the nature of activated complexes, and detailed mechanisms of certain processes.

The usefulness of the book is greatly enhanced by problems at the ends of chapters, exceedingly complete specific references and a general bibliography as well, and indexes both by subject and by author. The style is simple and clear, and errors are very few. The book has already been widely adopted as a text and without doubt its use will become even more widespread. It is to be highly recommended to all teachers of chemical kinetics.

W. J. ARGERSINGER, JR.

UNIVERSITY OF KANSAS
LAWRENCE, KANSAS

● ESSENTIALS OF NUTRITION

***Henry C. Sherman* and *Caroline Sherman Lanford*, Columbia University. Third edition. The Macmillan Co., New York, 1951. ix + 454 pp. 36 figs. 42 tables. 14.5 × 21.5 cm. $4.25.**

ALTHOUGH obviously designed as a text for use by classes, this book is useful as general literature and makes entertaining reading for anyone interested in health promotion by good nutrition. It includes the latest information on human nutritive requirements: calories, the more important inorganic elements, the better known vitamins, and the protein requirement. The authors frankly state that the book is not concerned with technical information on the chemistry of food and nutrition. It does supply, however, the information required for intelligent selection of a good diet.

The exercises at the ends of the chapters, together with wisely selected reading lists, make it possible for a student to have a good course in practical nutrition through the use of this book. It also gives an adequate treatment of food economics which should be of genuine aid to those who must be closely budgeted in food purchases. Extensive tables in the appendix give the composition and nutritive values of foods and recommended daily allowances.

Professor Sherman's long experience and ripened knowledge in the fields of food chemistry and of nutrition make especially valuable the first chapter, which assays the general significance of nutritional science in human welfare, and also the last chapter entitled How to Make Nutritional Knowledge More Effective, with subtitle, Nutrition Policy—Public and Personal. If nutritional education as here advocated could be promoted and given practical application, real benefit of nation-wide scope would result.

PHILIP H. MITCHELL

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● STRUCTURAL INORGANIC CHEMISTRY

***A. F. Wells.* Second edition. Oxford University Press, New York, 1950. xix + 727 pp. 237 figs. 82 tables. 16 × 25 cm. $7.**

SINCE the introduction of crystal structure determinations by the Braggs in 1913, the pursuit of the structural aspects of chemical compounds has become an integral part of the chemical approach to any problem. Hence it is important to have available a book which incorporates structural knowledge into the total fabric of chemistry. Of the various attempts to accomplish this objective, the one under consideration has been the most successful.

The first edition of this book was received very enthusiastically. Teachers of inorganic chemistry adopted it as a textbook. Reviewers commented favorably on the clear exposition of general topics without the use of advanced mathematics or thermodynamics, on the many excellent figures designed to aid three-dimensional visualization, on the excellent indexes and on the portrayal of unsolved problems.

The second edition retains all of the excellent features of the first and embodies a number of distinct improvements. The extent of the revision is evident by the fact that the first edition had 590 pages, 175 figures, and 31 tables.

The first part of the book deals with general principles and techniques. There are chapters on the covalent bond, interbond angles and bond lengths, ionic crystals, states of aggregation, the crystalline state, and experimental methods. Half of these have been completely rewritten and all have been amplified.

The second part presents a wealth of structural data which is organized under the following headings: hydrogen compounds, the halogens and simple halides; complex, oxy-, and hydroxyhalides; oxygen and sulfur; oxides; oxysulfides and sulfides; hydroxides; water and hydrates; nitrogen; phosphorus and Group V elements; carbon; cyanides, carbonyls, and carbides; silicon; boron; B subgroup metals; Group VII metals; *4f* and *5f* elements; and metals and alloys. Here again the material has been reorganized and considerably amplified.

References in the second edition are five times as numerous and more conveniently located than previously. Many of them bear dates of 1948 and 1949 showing that the revision is as up to date as a publication schedule permits. The formula index has grown from 5 1/2 to 10 pages and the subject index from 4 1/4 to 7 1/4 pages.

This book is highly recommended.

W. CONARD FERNELIUS

THE PENNSYLVANIA STATE COLLEGE
STATE COLLEGE, PENNSYLVANIA

● ORGANIC CHEMISTRY

***I. L. Finar*, Senior Lecturer in Organic Chemistry, Northern Polytechnic, Holloway, London. Longmans, Green and Co., New York, 1951. xv + 696 pp. 27 figs. 10 tables. 16 × 25 cm. $7.50.**

IN SOME ways this is just another textbook of organic chemistry, but it does have some unusual features and is well worth being brought to the attention of teachers in the field.

The arrangement of subject matter is in the "classical" tradition. Even the introductory one page of history and three pages of analysis and molecular weight determinations belong to the older school. The second chapter, called Structure of the Atom, is classical only in title, and serves as an excellent introduction to resonance, hydrogen bonds, hybridization of bonds, and reaction mechanisms. Then follow groups of sixteen chapters on aliphatic compounds, one on alicyclic, ten on aromatic, one on heterocyclic, and one on dyes. The space allotment seems wise and it is especially good to have the alicyclic compounds placed where they are, and to have their treatment include both the Baeyer Strain Theory and the Theory of Strainless Rings. In the reviewer's opinion, the reading references are more valuable than the questions at the ends of chapters. The latter are more nearly the common garden variety, while the references include all the fundamentally important books on each subject as well as recent journal articles.

One whole chapter is entitled Tautomerism, while no such prominence is given to the subject of isomerism in general. Actually, however, tautomerism shares its space generously, as would be expected, with the acetoacetic ester and malonic ester syntheses, hydroxyaldehydes and hydroxyketones, and aldehydic and ketonic acids. At the same time, stereoisomerism, both optical and geometric, occupies a large share of the chapter on hydroxyacids and unsaturated acids. The treatment of each subject is very complete and the accompanying chemistry in each chapter serves as the best possible illustrative material.

The electronic theories of valence introduced in Chapter II are used consistently throughout the book. Rather unusual in organic textbooks is their extension to include the molecular orbital theory. The diagrams are excellent for its application both to benzene (p. 410) and to butadiene (p. 67), the latter being used to illustrate Coulson's "delocalisation energy" ("but it is more usual to call it resonance energy") and for explaining 1,2- and 1,4-addition. Two further unusual and interesting features of the book are (1) the list of 50 English examination questions (pp. 660–2) chosen at the level of the B.Sc. General Honors degree and Part I of the Special Honors degree of London University; and (2) the unusually good discussion (pp. 664–70) of organic chemistry publications and the methods of using them.

The treatment of organic nomenclature is the least satisfactory feature of the book. It would be somewhat confusing to an American, since the English method is naturally used whenever a difference exists. The author chooses, for example, consistently such names as butan-2-ol rather than 2-butanol or butanol-2. Particularly awkward is "but-2-ene" (p. 63). However, even the differences and awkwardnesses might make the American student realize more fully the present state of flux of the subject, and it certainly does no harm to get the English point of view!

On the whole it is a solid, all-inclusive, well-written, well-printed, well-illustrated textbook, including both classical and modern viewpoints. It seems to fulfill the author's hope when he says he has "included detailed discussions on developments of a straightforward nature and also of a controversial nature, in the hope of encouraging the student to weigh up the evidence for himself."

HELEN S. FRENCH

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ADSORPTION AND CHROMATOGRAPHY

Harold Gomes Cassidy, **Associate Professor of Chemistry, Yale University. Volume V of "Technique of Organic Chemistry," edited by *Arnold Weissberger*. Interscience Publishers, Inc., New York, 1951. xix + 360 pp. 53 figs. 54 tables. 16 × 24 cm. $7.**

In the preface, the author has stated: "It is the objective of this book to describe and explain, by discussion of principles, the various methods by which adsorption may be utilized in organic chemistry as a tool for the separation of mixtures." To fulfill this objective the author has "not only describe(d) the various ways in which adsorption may be used, but also (has) explain(ed) the nature of adsorption and of the phenomena associated with it."

This book contains a great deal of information on adsorption gathered from the literature, which will be of aid either directly or by analogy to the chemist who desires to use adsorption as a method of separation. These data do not represent a compilation comparable to tables of solubility or boiling points which are so useful in designing separation methods based on precipitations and distillations. There is not at present a full enough body of knowledge about adsorption to permit such a tabulation.

In the chapter on the Nature of the Separation Processes, the author compares adsorption with other methods of separation which are based on the distribution of a substance between phases. No attempt is made to discuss a large number of phase-pair distributions but the examples illustrate the "similarities in principle and differences in application" of these processes.

Various types of adsorption methods, including Batchwise Adsorption and Decolorization, Chromatography, Ion Exchange Methods and Partition Chromatography, are discussed. A great deal of valuable information is compiled in tabular and graphical form in these chapters. The nature of the processes and the theory are discussed briefly with adequate reference to more complete sources. The apparatus and materials required and the manipulations necessary to affect the separations are discussed adequately. More detailed descriptions of procedures for carrying out several typical separations are given in the final chapter.

As the author states further in the preface, "Emphasis on the needs of the organic chemist has limited the discussion almost entirely to the properties and uses of liquid-vapor, liquid-liquid and liquid-solid interfaces. . . .

"In his 'Annotated Bibliography of Solid Adsorbents,' which covers the literature up to 1942, Deitz has listed some six thousand references. This represents only part of the literature in the much larger field of adsorption, and only someone who has attempted to read this literature can realize what an appalling task it would be to cover it completely. . . I have. . .tried to cover a wide variety of phenomena and to show their connections with adsorption."

To review this field is a huge task and in my opinion the author has done it well.

EDWARD R. TOMPKINS

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THE IDENTIFICATION OF MOLECULAR SPECTRA

R. W. B. Pearse, **Assistant Professor and Reader, Imperial College, London, and *A. G. Gaydon,* Warren Research Fellow of the Royal Society, Imperial College, London. Second revised edition. John Wiley & Sons, Inc., New York, 1950. xi + 276 pp. 12 plates. 20 × 28 cm. $8.50.**

The second edition of Pearse and Gaydon's excellent compilation of data on molecular band spectra is larger than the 1941 edition by some fifty pages. New data up to 1947, with some data and references as late as 1949, have been added and in many cases the rearrangement of the tables in more compact form has made possible a considerable increase in the amount of material included. "Four new plates containing 26 enlargements have been added, the new spectra shown including systems of O_2, various hydrides and other molecules of general interest."

As in the first edition, the compilation of available data has been limited to include "all recorded systems of diatomic molecules, but only those of triatomic and more complex molecules which show well-defined banded structure and are of frequent occurrence in spectroscopic investigations." Data on complex organic molecules and on solutions have not been included nor is there any discussion of the theory of molecular spectra but these omissions do not detract from the usefulness of the book in its fundamental purpose of "facilitating the identification of molecular spectra." The spectral region covered is, in general, from 10,000 Å. to 2000 Å. In the case of the absorption spectrum of molecular oxygen however, data are given as far as Å 1767.

Although the book is essentially a compilation of data from original papers, the authors have, in a number of cases, extended the recorded data by their own measurements. Their wide knowledge and experience in the field make their critical selection of material from the literature particularly valuable. The data are presented in two sections: "The first section consists of a list of the strongest heads of the more persistent and better known band systems of each molecule in order of wave length, together with information as to origin, intensity in various sources, and appearance. The second section consists of individual lists of band heads for each system of each molecule, accompanied by notes about the occurrence and appearance of the system, the nature of the electronic transition involved, the vibrational assignment of the bands in the system, and references to the sources of the data."

The section called "Practical Hints" was an extremely useful feature of the first edition; this has been retained, with some additions, in the new edition. This material is not ordinarily available in textbooks and gives invaluable help to the person inexperienced in the identification of molecular spectra. The number of scientists who are concerned with the identification of band spectra is probably not great, but certainly this book would be indispensable to those who are working in this field. In the words of the authors, "such a list can be of service not only to pure

spectroscopists, but also to those who use spectroscopy as a tool for research in other fields such as astrophysics, chemistry and chemical technology."

The authors are to be congratulated on the care and skill with which they have accomplished their task and the publishers should also be commended for the excellent form in which this second edition is presented. In these days of paper shortage and publishing costs there is a frequent sacrifice of form to expediency. Such is not the case with this book; the spectrograms are beautifully reproduced and the printing and arrangement of the data are all that could be desired.

EMMA P. CARR

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South Hadley, Massachusetts

● METHODS OF SYNTHESIS IN ORGANIC CHEMISTRY

John H. Billman, **Associate Professor of Chemistry, Indiana University, and** ***Elizabeth S. Cleland,*** **Former Instructor of Chemistry, Goucher College. Second edition. Edwards Bros., Ann Arbor, Michigan, 1950. viii + 229 pp. 14.5 × 22.5 cm. $2.75.**

This book is intended as a "study guide," or as a supplement to a textbook, for students of elementary organic chemistry. It is not in any sense a laboratory manual.

The 15 chapters are arranged as outlines to include what the authors feel are "the more common standard basic reactions." The reactions are classified in the usual manner as they pertain to the common type compounds. Each chapter is divided into four parts: nomenclature, preparations, properties, and problems. Separate chapters on the preparation and properties of the halides and on the methods of varying chain lengths are included to serve as review. Aliphatic and aromatic compounds are taken up together.

The problems at the end of the chapters are designed primarily to test the student's ability to name structural formulas, to write structural formulas when given systematic names, and to outline methods of synthesis for specific compounds. The answers to the problems are placed in a separate section and occupy a large portion of the book. Brief comments concerning the syntheses are inserted with the answers where they appear pertinent. In some cases more than one synthetic route is noted, but no attempt is made to indicate all possible routes.

Many students of elementary organic chemistry should find this book quite useful. The authors' suggestion that the format of the outlines may be used as a pattern by which to outline lecture notes seems logical. Considering the two extremes, a bare compendium of reactions and a reasonably complete textbook, this book is a respectable compromise.

DONALD C. GREGG

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● LIQUID EXTRACTION

Robert E. Treybal, **Professor of Chemical Engineering, New York University. McGraw-Hill Book Co., Inc., New York, 1951. vii + 422 pp. 262 figs. 22 tables. 15.5 × 24 cm. $7.50.**

"In distillation processes, a mixture of two substances is separated by creation of two phases, one liquid and one vapor, by the addition of heat, and the separation is brought about by virtue of the fact that the relative concentration of the substance is different in the two phases. Subsequent condensation of the vapor phase is brought about by removal of heat. In extraction, two liquid phases are formed by the addition of an immiscible solvent, which then becomes the analog of heat." This distillation analogy is used successfully by the author in tying much of the theory and calculation to more familiar distillation phenomena.

The book can be divided into four sections: basic theory (124 pages), methods of calculation (132 pages), equipment for extraction (89 pages), and liquid extraction processes (53 pages). After a brief introduction as the first chapter, the second chapter gives a complete discussion of liquid equilibria from the viewpoint of the phase rule; methods of graphical representation are shown for ternary and quarternary systems as well as tie line correlations. For the prediction of distribution (Chapter 3) the Gibbs-Duhem equation is integrated and the Wohl equation used to give simple equations to reproduce activity coefficients for binary systems and calculate activities for ternary systems. The choice of solvent is discussed in Chapter 4. The rate of attainment of equilibrium is contained in the fifth chapter of diffusion and mass transfer. The second section on methods of calculation divides these into stagewise contact with a single solvent (Chapter 6), stagewise contact with mixed and double solvents (Chapter 7), and continuous countercurrent contact (Chapter 8). Equipment is divided into that for stagewise contact (Chapter 9) and for continuous countercurrent contact (Chapter 10). The final chapter (11) describes liquid extraction processes used in various industries.

The book is thoroughly up to date and each chapter is concluded with a list of literature references used in that chapter, references which include papers published in 1950. The lists are extensive and enable the reader to refer to the original literature. References to foreign language papers are used infrequently, which is satisfactory in a textbook. The appendix contains a number of problems for most of the chapters in the book. The material is presented clearly and logically. The figures are all diagrammatic and are drawn to show certain points which the author desires to present, or to illustrate graphical methods of calculation.

"Liquid Extraction" is a welcome addition to the texts available for graduate instruction in chemical engineering. The material is presented in a fundamental manner so that the chemist interested in extraction processes, either for the laboratory or for the plant, will find material of interest and importance to him.

KENNETH A. KOBE

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● THE CHEMISTRY OF THE NONBENZENOID HYDROCARBONS

Benjamin T. Brooks, **Consulting Chemist, New York. Second edition. Reinhold Publishing Corp., New York, 1950. xiv + 615 pp. 31 figs. 78 tables. 16 × 23.5 cm. $12.**

The second edition of this well-known work has long been due. The first edition, published in 1922, had fallen far behind the theoretical and technological aspects of the field. Dr. Brooks' new edition summarizes the latest advances in the chemistry and applications of nonbenzenoid hydrocarbons and of their closely related derivatives.

The book deals with all types of nonbenzenoid hydrocarbons—paraffins, cycloparaffins (including terpenes), olefins, and acetylenes. Methods of synthesis, properties, and reactions with common chemical agents are discussed in detail for each class. Important industrial processes such as thermal cracking, oxidation, hydrogenation, polymerization, alkylation, isomerization and the oxo process are presented with clarity. Topics such as the relationship of antiknock properties to structures of compounds, natural and synthetic rubber, the Prins reaction, and theory of engine knock are covered with a great deal of understanding.

Other valuable works such as those of Ellis and Egloff have covered somewhat the same areas, but not in the same manner. Apparently no attempt was made here to record all published papers and patents on each subject. Very careful selection of material has been made so that the reader obtains a well-rounded idea of the complete field. Enough references to the original literature are given so that one may follow up a particular line of interest without difficulty. The book is well written, printed, and

indexed, so that the reader can easily locate and utilize the information desired.

This reviewer would have liked a little more attention given to nomenclature, but otherwise no adverse criticism should be made.

The book will be of interest to workers in several fields. Specialists in one branch of hydrocarbon chemistry will be able to keep up with developments in other branches. Organic chemists and chemical engineers in other fields will find this a valuable source of information concerning hydrocarbons and their simple derivatives. Students and teachers will find themselves turning to this book on many occasions for information concerning nonbenzenoid hydrocarbons.

ERNEST M. HODNETT

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THE ANALYTICAL BALANCE: ITS CARE AND USE

***William M. MacNevin,* Professor of Analytical Chemistry, The Ohio State University. Handbook Publishers, Inc., Sandusky, Ohio, 1951. xiii + 60 pp. 12 tables. 13.5 × 20.5 cm. $1.50.**

THIS little book, which does not discuss the theory of the balance and contains neither drawings nor diagrams, is written to provide the practical information needed to care for and use the analytical balance. It is stated that the book "is designed to acquaint the average scientific worker with the problems of selecting, mounting, cleaning, adjusting, testing, and repairing a balance." However, it is recommended that major balance repairs be done by a specialist. To this end a list of about twenty Balance Repair Specialists and their addresses is given. In the introduction, tables of analytical balances in use in American laboratories, and of manufacturers of analytical balances are given. The text first inventories the parts of a balance; then gives the details of mounting, cleaning, and assembling; then provides elementary instructions in using the balance; then follows with tests for ascertaining the condition of the balance. The various classes of analytical weights are next considered and detailed instructions given for the calibration of weights, beam notches, and chain. The systematic application of weight calibration corrections is considered and also methods for actually adjusting the masses of the weights to the required tolerances. In considering the buoyancy correction in weighing it appears that the author might well have taken occasion to point out the relative merits of true mass as compared to apparent mass versus brass in assigning absolute weight calibrations, as this question arises in the use of a Bureau of Standards certificate.

This practical little manual may well find a useful place in many commercial and technical laboratories.

W. W. RUSSELL

BROWN UNIVERSITY
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PROGRESS IN CHROMATOGRAPHY 1938–1947

***L. Zechmeister,* Professor of Chemistry, California Institute of Technology. John Wiley & Sons, Inc., New York, 1951. xviii + 368 pp. 23 figs. 21 tables. 14 × 22 cm. $8.**

IN 1906 the Russian botanist Michel Tswett separated a mixture of leaf pigments by adsorption of an extract of green leaves in a deep, porous bed or column of powdered absorptive chalk. To effect this separation, he filtered a small quantity of the pigment mixture into the uppermost portion of the column. He then washed the absorbed mixture with fresh solvent so that the pigments migrated through the column at different rates and formed a series of colored zones each of which contained a single component of the mixture. This formation or development of the chromatogram by washing with fresh solvent is the unique feature of the chromatographic adsorption method, now widely utilized for the resolution of all kinds of soluble substances.

Professor Zechmeister was among the first to recognize the high resolving power and the wide applicability of Tswett's analytical method. His first book in this field, written with L. Cholnoky in 1937, revised in 1938, and translated into English by A. L. Bacharach and F. A. Robinson in 1941, has contributed enormously to the utilization of chromatography as a common analytical tool.

The current book, which is devoted to a review of the literature for the years 1938 to 1947, is, essentially, a supplement to the earlier publications. It surveys virtually all the new techniques, principles, and applications of chromatography during this period. By far the greater portion of the book (pages 60-285) describes the chromatographic examination of various substances ranging from chlorophylls to nitrogen isotopes. A bibliography of 40 pages and comprehensive author and subject indexes complete the volume.

The value of this book to students and teachers will depend upon their interest and experience. As most of the basic chromatographic principles were included in the first publication, this new volume will be of greatest aid to workers with some experience and to those already familiar with the first volume. It will be especially useful as a guide to workers seeking specialized information about the separation of mixtures of particular substances.

HAROLD H. STRAIN

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BIOCHEMISTRY OF GLUCURONIC ACID

***Neal E. Artz* and *Elizabeth M. Osman,* Corn Products Refining Co., Argo, Illinois. Academic Press, Inc., New York, 1950. vii + 103 pp. 15.5 × 22.5 cm. $2.50.**

THE purpose of this book, as stated by the authors in the preface, is to bring together the widely scattered observations bearing on the metabolic significance of glucuronic acid. This acid is available now in adequate quantities. It is the hope of the authors that this compilation may be of service to investigators who wish to do work involving glucuronic acid.

The extent to which the literature of the last hundred years, since the discovery of glucuronic acid, has been examined is indicated by a bibliography of 456 references. The book has been divided into four parts, reviewing the literature of analytical methods, occurrence, origin of glucuronic acid in the body, and detoxification. By far the largest portion of the book is devoted to the role of glucuronic acid in detoxification. In this part are listed the large number of substances that are conjugated with glucuronic acid after administration to animals and excreted as glucuronosides.

Presumably the coverage of the literature is fairly complete. However, with the exception of the chapter on analytical methods, this review offers little in the way of critical evaluation of the experimental work involving glucuronic acid. The reader will look in vain for an adequate discussion of the literature of glucuronic acid metabolism from the point of view of indicating the problems that future research on this interesting biological compound might pursue with profit.

F. A. CAJORI

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A SYSTEMATIC LABORATORY COURSE IN GENERAL CHEMISTRY

***Harry H. Sisler,* and *Jay J. Stewart,* The Ohio State University. The Macmillan Co., Inc., New York, 1950. xi + 396 pp. 57 figs. 21 × 27.5 cm. $3.**

THE intention, stated by the authors, of creating a laboratory manual that could be used by students having different backgrounds and would provide factual chemistry, enlargement of the

understanding of the physical world, and development of the ability to apply the scientific method has been fulfilled by a collection of 90 experiments. These have been grouped into 20 sections each containing related material which provide a choice of several experiments for each of the general subjects studied in an elementary chemistry course. Eleven appendixes containing all of the usual tables and directions for making up the solutions make the manual very complete.

The traditional approach to qualitative analysis has been abandoned in favor of studying the properties of the elements in groups as suggested by the periodic table and then providing for the application of this information by analysis of simple salts and mixtures as chosen by the instructor. Each experiment contains blank spaces for fill-in answers and each may be detached and turned in for grading purposes.

The all-purpose nature of this manual coupled with the ease of grading, makes it a good choice for large classes. It would be especially useful where the larger class could be subdivided into groups with the same background, general ability, or need for a type of preparation in elementary chemistry.

JAMES F. CORWIN

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YELLOW SPRINGS, OHIO

● SIMPLIFIED CHEMISTRY EXPERIMENTS

Armand Joseph Courchaine, **Instructor in Biological Chemistry, Hahnemann Medical College, Philadelphia, Pennsylvania. G. P. Putnam's Sons, Inc., New York, 1950. xxi + 234 pp. 13 figs. 21 × 27.5 cm. (Spiral bound.) $2.80.**

THIS manual is designed for use with a textbook that places more emphasis on organic and physiological chemistry than is usual in the average elementary course. Each experiment contains blank spaces for fill-in type answers and no extra notebooks or papers are necessary. The order of presentation of the material follows that of the author's own textbook, "Chemistry, Visualized and Applied."

Of the 26 experiments, the first 12 are inorganic and deal with the properties of material in solution, the remainder are concerned with material that is organic or physiological in nature. Teachers who have the problem of organizing the laboratory work for courses in chemistry, for nurses, for laboratory technicians or for home economics students, would find this manual useful.

JAMES F. CORWIN

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● EXERCISES IN GENERAL CHEMISTRY

Harold G. Dietrich, **Assistant Professor of Chemistry, and** ***Erwin B. Kelsey,*** **Associate Professor of Chemistry, Yale University. The Macmillan Co., Inc., New York, 1950. x + 285 pp. 18 figs. 21 × 27.5 cm. $3.**

THIS collection of 43 exercises represents a larger number of actual experiments. Elements having the same general properties are studied as a group rather than separately, and experiments illustrating the same general principle are found in the same exercise. Report blanks that may be detached accompany each of the quantitative exercises but no fill-in reports are included. The right-hand sides of most of the pages in the book are left blank for writing up the experiment in the form suggested by the individual instructor. Six appendixes provide the necessary tables and directions for solution preparation.

The experimental material in this manual covers all of the usual work in general chemistry, and the explanations of the procedures are designed for use even with students having no previous laboratory experience. Selection of experiments within an exercise rather than selection of exercises offers comprehensive coverage of the laboratory work. Qualitative analysis as a subject has been integrated into the study of the elements and ions as groups rather than the traditional approach.

Teachers who favor the written report and who wish a complete coverage of elementary chemistry in the laboratory will find this manual helpful.

JAMES F. CORWIN

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● COLLEGE CHEMISTRY IN THE LABORATORY

Lloyd E. Malm, **Professor of Chemistry, University of Utah, and** ***Harper W. Frantz,*** **Professor of Chemistry, Pasadena City College. W. H. Freeman and Co., Inc., San Francisco, 1950. xxxvi + 331 pp. 93 figs. 21.5 × 27.5 cm.**

THIS manual which contains 46 experiments is designed to accompany Pauling's "College Chemistry," and references are made to particular chapters in the textbook to which each experiment applies. Four extensive appendixes cover all necessary tables, methods of calculation, and directions for preparation of experiments. Laboratory safety is emphasized by numerous, well-chosen illustrations. These illustrations, along with graphic representation of chemical principles and illustration of laboratory setups, make this manual outstanding.

Although the manual is written to accompany a particular book, the material covered is very similar to that which appears in most general chemistry textbooks and, with proper selection can be made to fit any course. Teachers who like a different and more modern approach will find that this book meets their expectations.

JAMES F. CORWIN

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● FUNDAMENTALS IN CHEMISTRY FOR THE LABORATORY

W. Conard Fernelius, **Professor of Chemistry, The Pennsylvania State College;** ***Alfred B. Garrett,*** **The Ohio State University; and** ***Laurence L. Quill,*** **Professor of Chemistry, Michigan State College. Ginn and Co., Inc., Boston, 1950. Revised edition. viii + 367 pp. 103 figs. 21 × 26.5 cm. $2.75.**

THE 88 experiments collected into this book form a very complete coverage of general chemistry, but do not include any qualitative analysis. The experiments vary in complexity from simple test-tube experiments and exercise blanks to elaborate collections of apparatus. It is suggested that the more complex be done as classroom demonstrations. The fill-in type of answer is provided for throughout each experiment and the pages may be torn out for grading purposes. Five very complete appendixes provide tables and descriptions of the necessary solutions.

This manual offers not only a complete coverage of laboratory experiences, but also a selection of demonstration experiments that may be used in class. The procedures are designed in general for those students who have little or no laboratory experience. Teachers who prefer the traditional approach to general chemistry and like a complete plan of demonstration and laboratory experience for their students will find this manual useful.

JAMES F CORWIN

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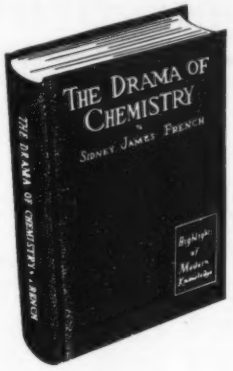

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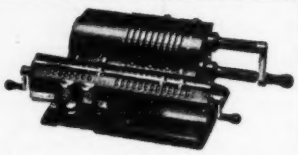

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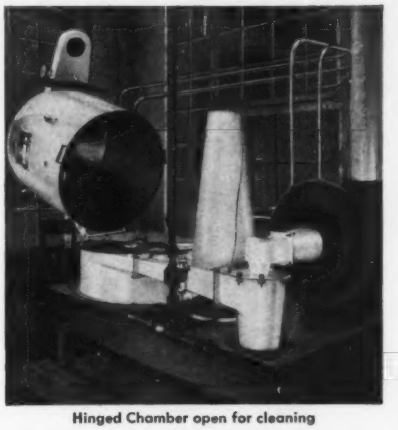

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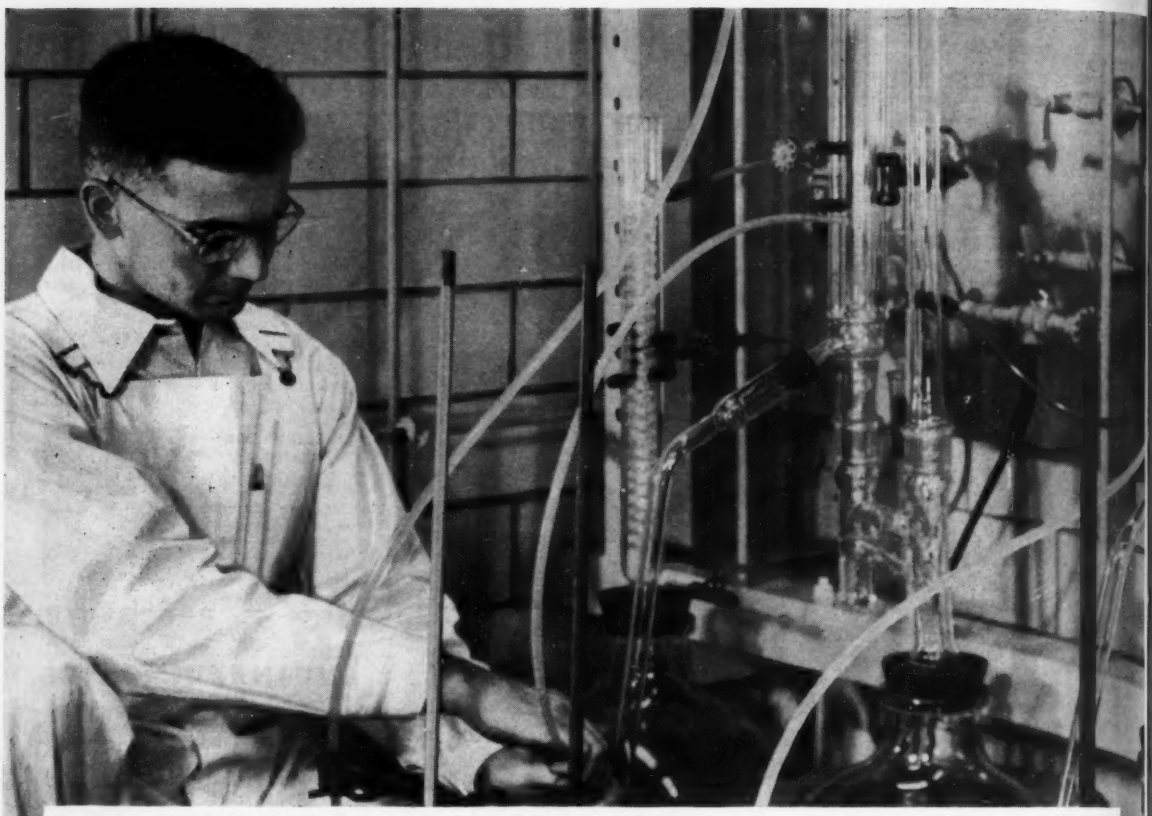

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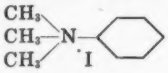

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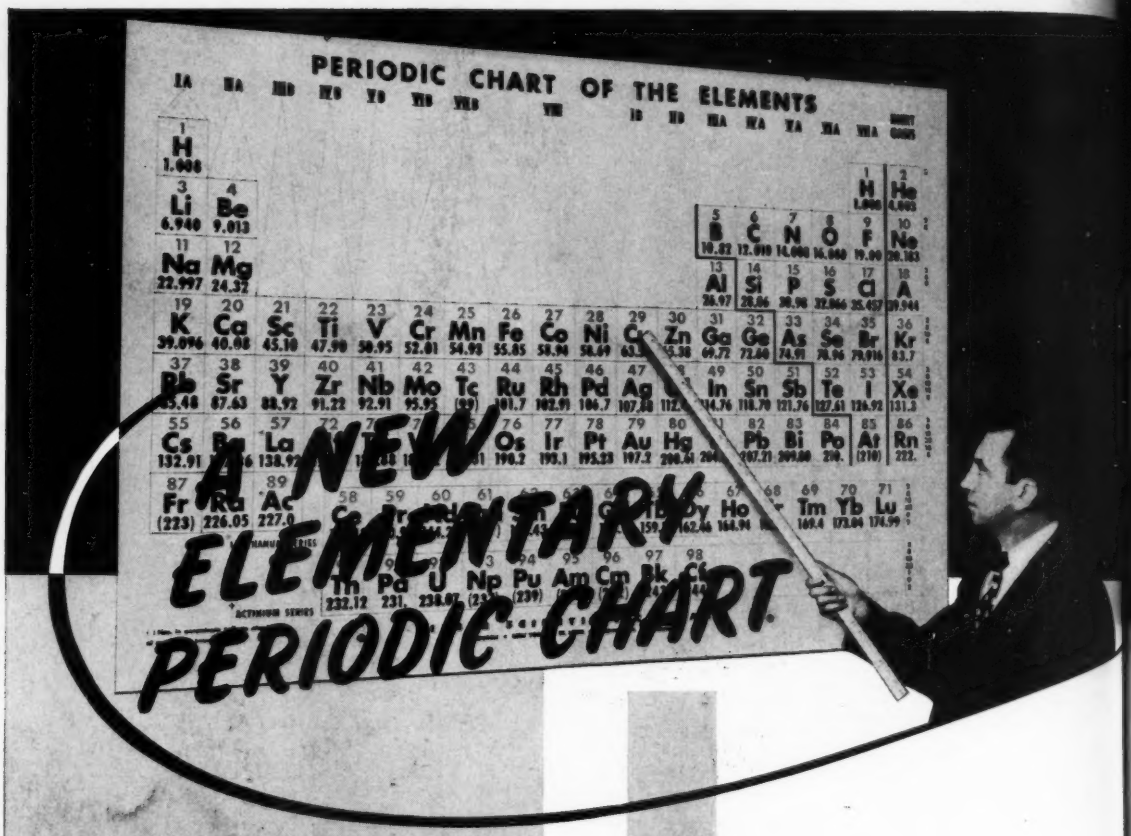

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